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A Study of the Thermal Mechanical and Micrographic Properties of Pure Iron

Electrical Engineering

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A STUDY OF THE THERMAL MECHANICAL AND MICROGRAPHIC PROPERTIES OF PURE IRON

BY

RUDOLPH McDERMET

B. S. University of Illinois, 1912

THESIS

Submitted in Partial Fulfillment of the Requirements for the

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I HEREBY RECOMMEND THAT THE THESIS PREPARED UNDER MY SUPERVISION BY

Rudolph McDermet

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BE ACCEPTED AS FULFILLING THIS PART OF THE REQUIREMENTS FOR THE

DEGREE OF Master of Science in Electrical Engineering

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I

INTRODUCTION

Since almost the beginning of the Christian era experience has been available in at least a limited way regarding the uses and mechanical properties of iron-carbon alloys; within recent years this information has been amplified and interpreted from a scientific standpoint, and the influence of other alloying elements has been studied until now ternary and quarternary systems are well understood. Because of the difficulty of producing iron in a pure and workable form little is known of its properties. It is the purpose of this thesis, therefore, to present a series of mechanical tests on pure iron, and to explain the results obtained in the light of modern methods of research and according to the latest theories.

Concisely it is the intention first to investigate the thermal critical points as a basis for regulating heat treatments; then to perform a series of tensile tests on specimens which have undergone these arbitrarily determined thermal treatments; and finally to correlate the mechanical tests with micro-photographic analysis in an endeavor to explain the results obtained. From this viewpoint the thesis may be logically grouped in three parts;

1. The Determination of the Thermal Critical Points.
2. The Results of the Mechanical Tests.
3. The Micro-photographic Analysis.

All of the data to be presented was available for bulletin 73 of the Engineering Experiment Station. Some of it has already been used for this publication and it was for this reason that the system used for designating specimens in the bulletin was retained in the thesis.

The method of preparation for the iron has been the subject of much difficulty and laborious research. It will be sufficient in this connection, however, to pass over the preliminary work and mention only the final method. Briefly the pure iron employed in this investigation is made from Swedish charcoal iron doubly electrolytically deposited on lead cathode plates from a ferrous sulphate solution, and subsequently remelted in vacuo in a carbon resistor Arsem vacuum furnace in contact with a magnesite crucible. In order to furnish a criterion of the behaviour of the pure iron, three alloys of varying carbon content were also prepared according to the method above, but with the addition of a known amount of granulated graphite carbon. A table of chemical analysis is appended as table 2 .

II

DETERMINATION OF THE CRITICAL POINTS

A very large amount of work has been done on the question of the allotropy of iron, and out of this mass of data the following facts have emerged. In the neighborhood of 900°C. iron undergoes an allotropic modification which is indicated by an evolution of heat on cooling and an absorption of heat on heating. This point is designated as the A_3 point, and is different for heating and cooling, and is lowered by the addition of impurities, particularly carbon. A similar and less pronounced point occurs around 750° C. Besides the changes in thermal equilibrium, marked changes in volume, specific heat, electrical resistance, thermo-electric force, and magnetic permeability have been noted. To account for these radical changes in critical properties the following theories have been formulated.

(1) The critical points A_3 and A_2 abruptly divide iron into three allotropic forms; gamma iron above A_3, beta iron between A_3 and A_2 ; and alpha iron below A_2 . This is the theory of Osmond.

(2) The point A_3 is an evidence of an allotropic change at its corresponding temperature, but the point A_2 is not, and is merely an evolution of heat accompanying a change in the magnetic properties.

(3) The critical point A_2 is not an evidence of an allotropic change but is merely a manifestation of the ending of the

transformation which began at A_3. This interpretation has behind it the prestige of Le Chatelier.

The last two hypotheses deny the existence of beta iron. In view of recent developments which indicate that distinct marked changes in all the properties of the metal with the possible exception of the crystalline structure actually occur at the A_2 point, credence is being given more and more to the theory of Osmond. It has been decisively proved that alpha and gamma iron crystallize in different systems, and it is difficult to assume that the two are mutually soluble, while there is considerable evidence in favor of Osmond's theory that alpha and beta iron are isomorphous. The work in this thesis will, therefore, be based on the validity of the first theory, and the method of investigation will be that of heat evolutions. The detailed statement of the procedure follows.

After melting and solidifying in the vacuum furnace the ingot was forged in an oxidizing coke forge into a long rod about one half inch in diameter. From this rod cooling and micro specimens were cut and the tensile test specimens lathe-turned. The cooling curve specimens were rarely more than fifty grams weight and usually were smaller. A small hole was drilled in them sufficient to enclose and protect the thermo-couple hot junction. The specimen was then ready for use.

In obtaining the cooling curve the specimen was placed in contact with the thermo-couple in an electro-quartz pyrometer tube partially filled with fused magnesite. Tube and contents

were then inserted in a Hoskins muffle type resistance furnace and heated approximately to 950° C. At this point current was cut off and furnace and specimen allowed to cool. Owing to the insulating properties of the magnesite the specimen was independent of the thermal conditions inside the furnace, while maintaining practically the same cooling rate. During the cooling the elapsed time for each successive ten degree decrement was taken by stop watches, one being stopped and the other started simultaneously as the magnified image of the pyrometer galvanometer needle passed over a scale division.

In all temperature determinations the same pyrometer was used. It consisted of a platinum-rhodium Haereus element standardized for use with a high resistance Siemens-Halske galvanometer. The pyrometer was frequently calibrated against the following fixed points with the cold junction at 0 C, and its indications were found to be remarkably uniform. A calibration curve for the instrument is included as page 16 .

Sn	232 C	
S	444 C at 760 mm.	
Al	658	
Ag	961	Reducing conditions
Cu	1084	Reducing conditions.

RESULTS

From the data obtained as described three curves were plotted on the same sheet and to the same scale of temperature

abscissae after the data had been suitably transformed by calculation:

(1) The temperature-time cooling curve with centigrade temperatures as abscissae and time of cooling in minutes as ordinates. This is the fundamental curve and should indicate allotropic transformation points by jogs. Since, however, its indications are not always decisive, the other two curves are included.

(2) The inverse-rate curve plotted with the time in seconds for the occurence of a ten degree decrement as ordinates. This should indicate critical points by its maxima.

(3) The temperature rate curve plotted with the rate of cooling in degrees per minute as ordinates. This indicates critical points by its minima.

Previous to taking the cooling curves of the specimens, a series of cooling curves of the furnace were taken in the manner indicated. These curves are included to show the effect of the furnace upon the cooling rate.

The different critical points for the different specimens are included in table I. The following remarks are significant, however, in understanding the results of the determinations.

The sensibility of the method is plus or minus five degrees. From the inflection points on the curves and the experience gained in the manipulation it is the opinion of the writer that the Ar_3 (the A_3 point on cooling) is located very close to 895 C. In some cases it is possible to observe the hang of the galvanometer needle closer than the limit of accuracy

settled upon above. This observation was considered in locating the assigned value of the temperature for the point in question. Similarily the location of the Ar_2 point is decided upon as being approximately 760° C. An Ar_{3-2} point was detected only in the case of the carbon alloy 3-C0-3. An Ar_1 point was also located for this specimen, a phenomenon which is not to be expected for so low a carbon content.

Certain qualitative facts may also be gleaned from the results of these experiments.

The Ar_3 point is not to be confounded with the equilibrium point A_3. Preliminary experiments indicate that, for equal rates of heating and cooling as determined by the normal cooling rate of the furnace, the Ar_3 point and the Ac_3 point may be separated by as much as forty degrees. In the case of the Ar_2 point it is not supposed that it differs from the A_2 point by more than five degrees.

Previous thermal and mechanical treatment probably greatly affect the location of point Ar_3 ; the same is not true of Ar_2 which is relatively undisturbed by any previous treatment of the specimen. It is not in general possible to check values obtained for one trial by reheating the specimen, but the position of the point varies slightly with the rate of cooling and with the maximum temperature peak, and the unreliability increases enormously with the number of successive heatings. This is due probably to the presence of occluded gases.

The Ar_2 point is more sharply defined than the Ar_3

point, but the order of the magnitude of the heat evolutions is roughly as one to four. Each point is, however, definitely marked.

For the best results in making the determinations the specimens should be small and compact and of a weight not exceeding thirty grams. The larger specimens do not give as sharply located points as the smaller.

The method here employed is believed to be admirable in the location of critical points in the practical heat treatments of steel. It is efficient in time,labor and apparatus and does not require skillful manipulation. An improvement might be inferred along methods of heating and cooling in vacuo and the use of high sensibility auto graphic pyrometers.

Table 1

CRITICAL POINTS

Electrolytic Iron

	Ar_3	Ar_2
3-30	885	765
3-31	885	775
3-33	895	765
3-34	890	760
3-35	893	755
3-36	875	760
3-37	885	760
3-38 #1	905	785
3-38 #2	895	760
3-39	905	755
3-40	895	750
3-41	892	755
3-43	895	755
3-45	895	755
3-47	897	760
Average	892	761

Carbon Alloys

3-CO-1	895	755
3-CO-2	895	755
	Ar_{32}	Ar_1
3-CO-3	750	685

Table 2

CHEMICAL ANALYSIS

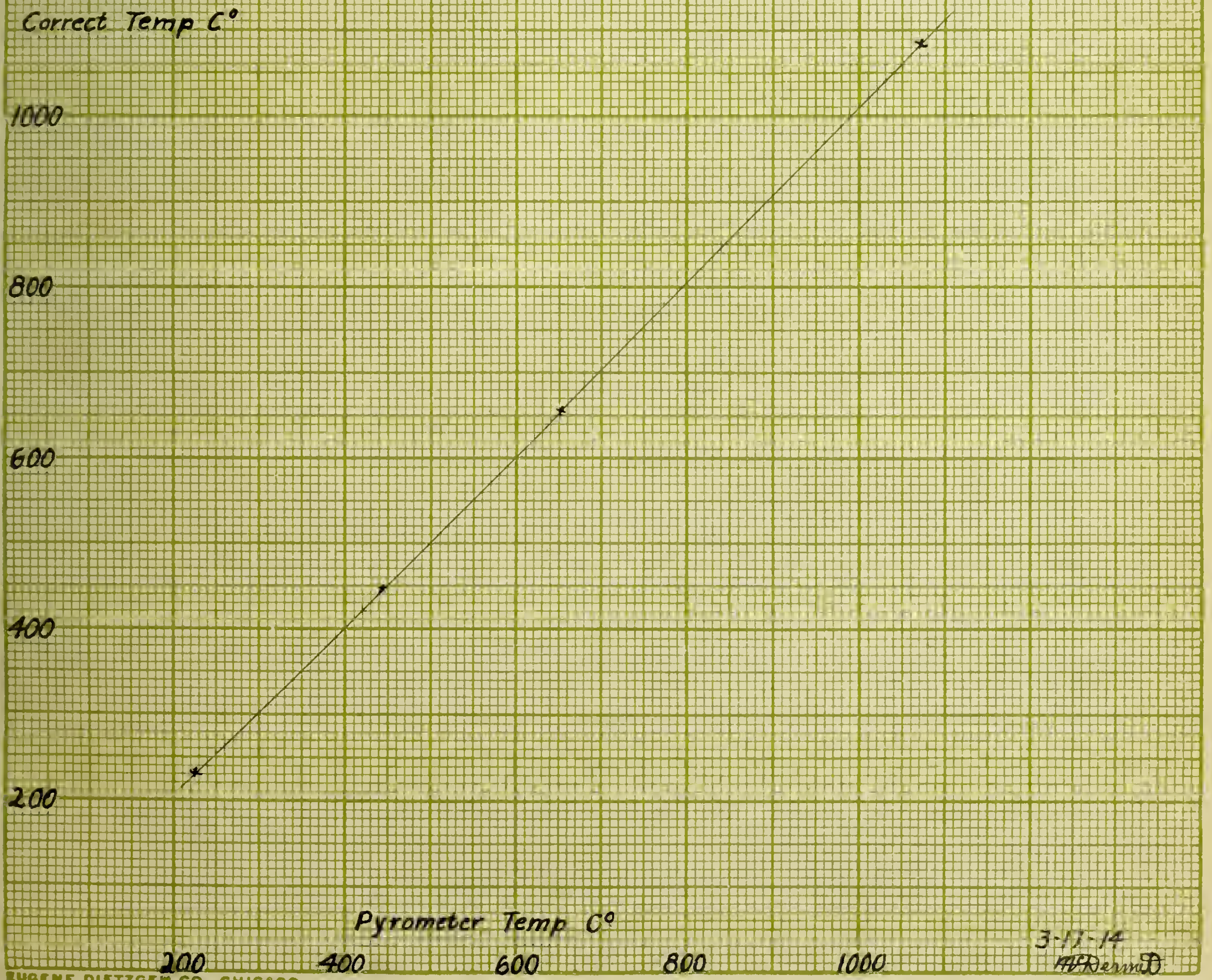
	Mn	P	S	Si	C
Swedish Iron	None	None	.0002	.032	.163
Electrolytic				.01	.006
Vacuum melted					
3-30					.009
3-31					.012
3-34					.009
3-36					.015
3-37					.045
3-38					.0104
3-39					.011
3-40					.011
3-41					.0095
3-43					.0196
3-45					.008
3-47					.008
3-48					.008
3-49					.012
3-50				.0015	.0099
Carbon alloys					
3-CO-1					.013
3-CO-2					.012
3-CO-3					.181

CALIBRATION CURVE

Haereus Pyrometer

Cold Junction 0 0°C

Sn	227	232
S	437	444
Al	651	658
Cu	1071	1084



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H.E. Dermod

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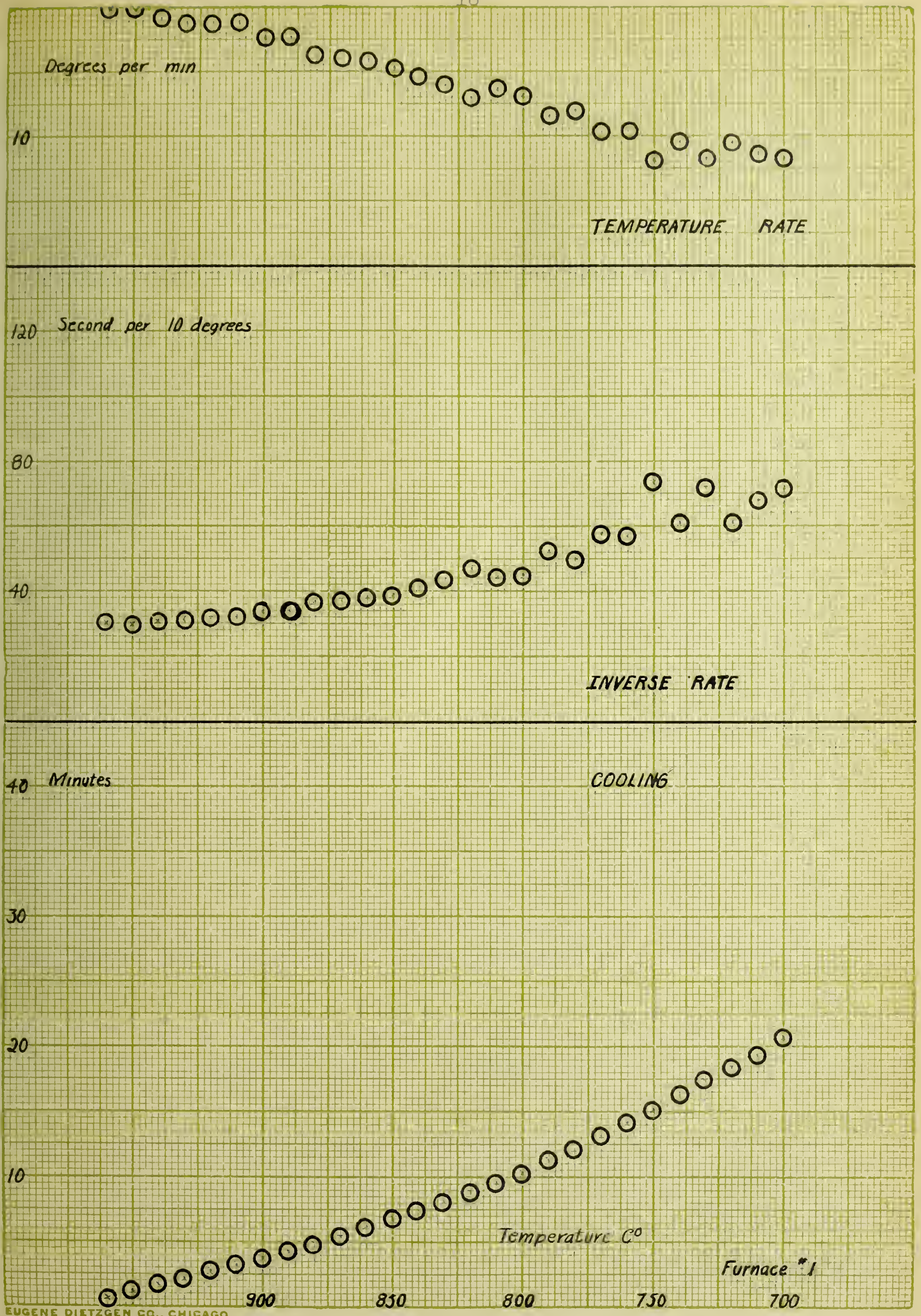

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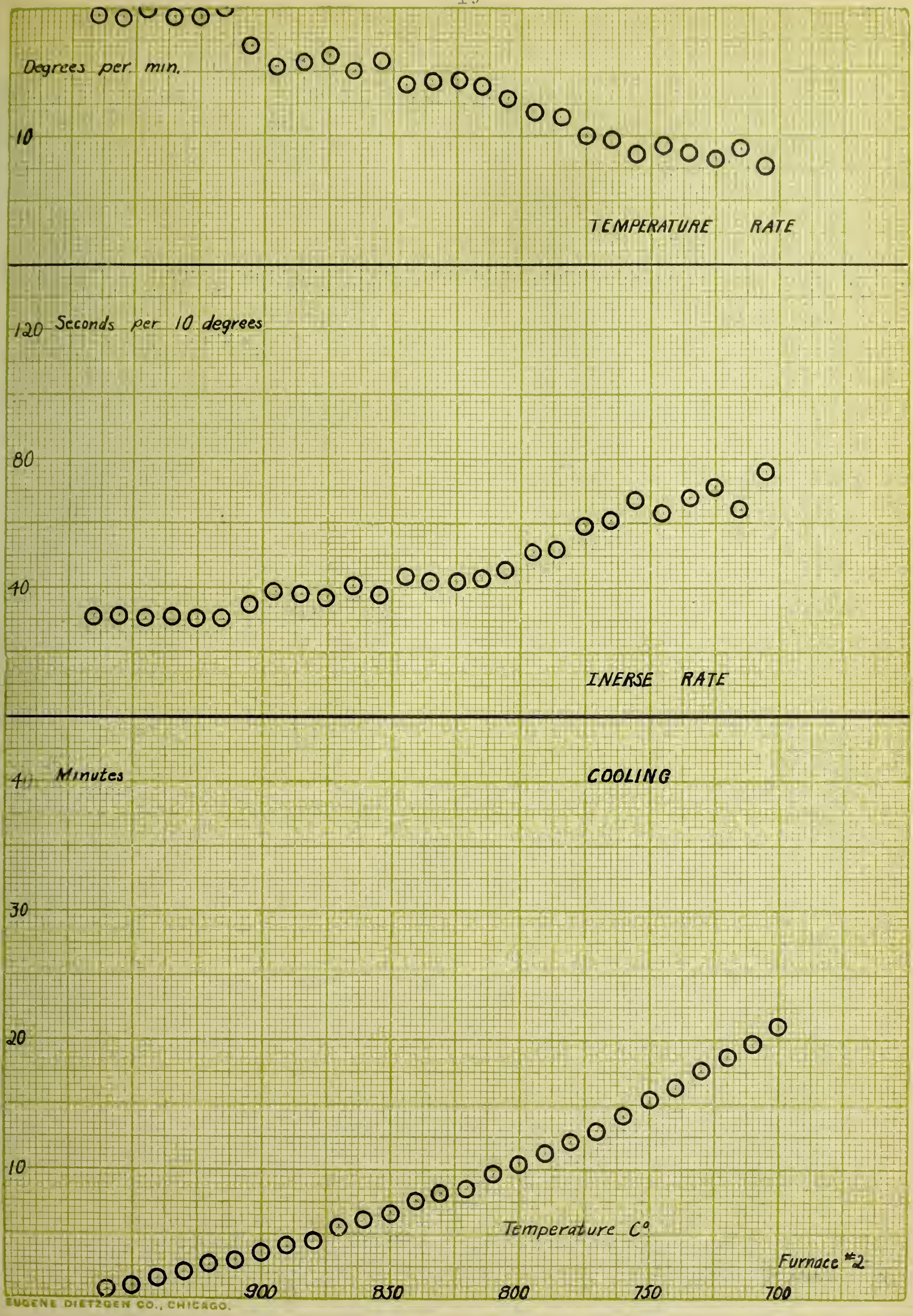
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Seconds per 10 degrees
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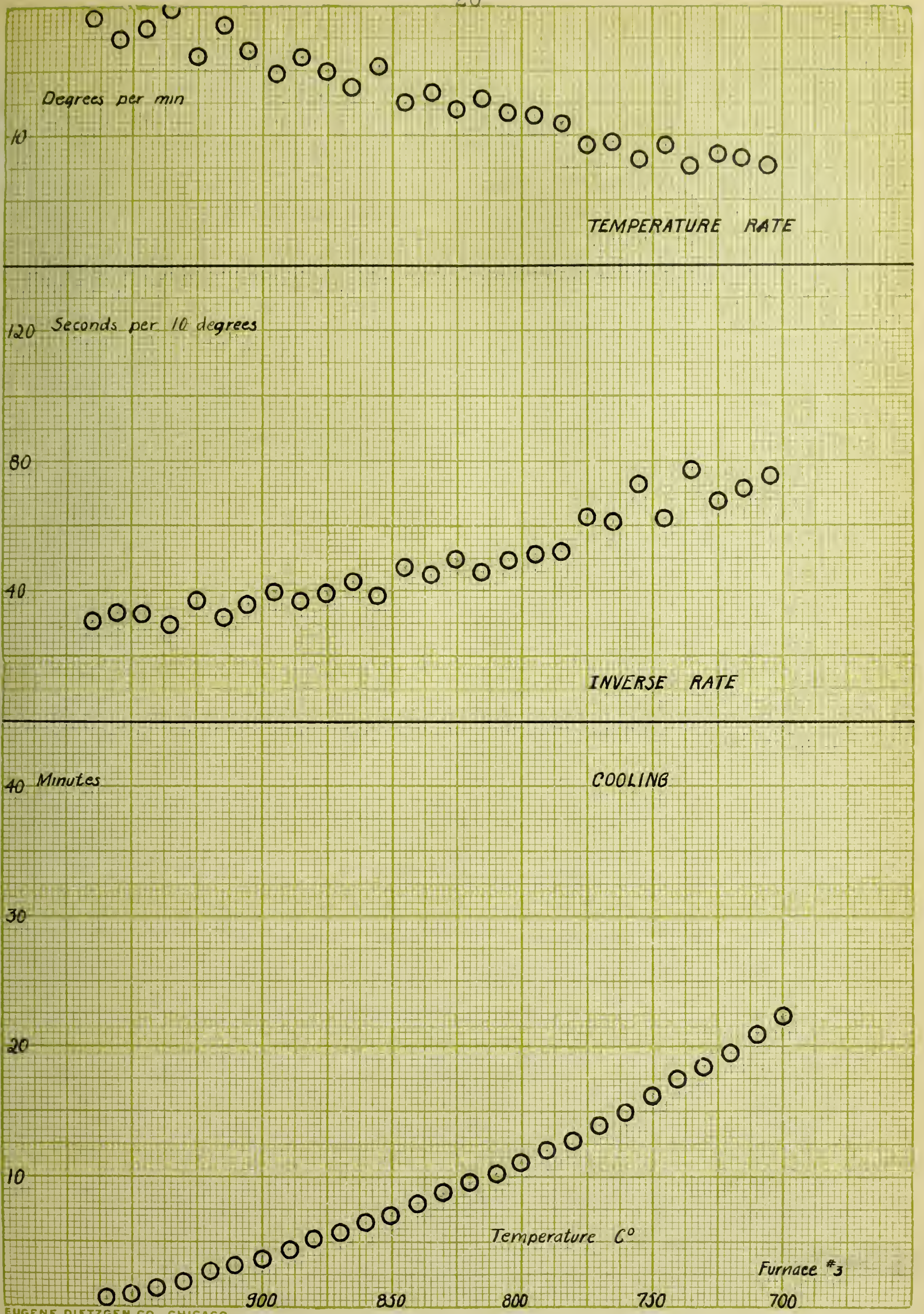

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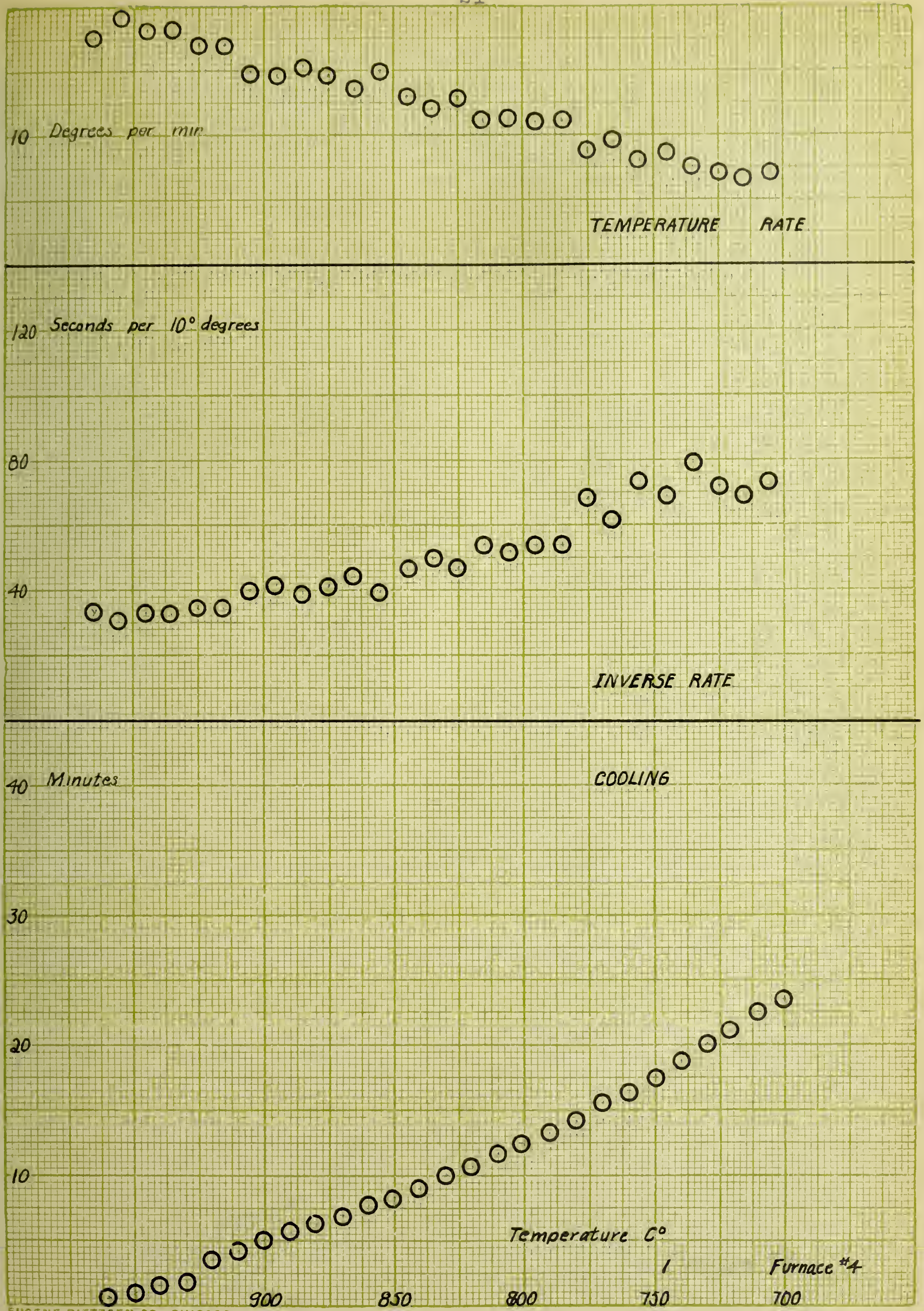

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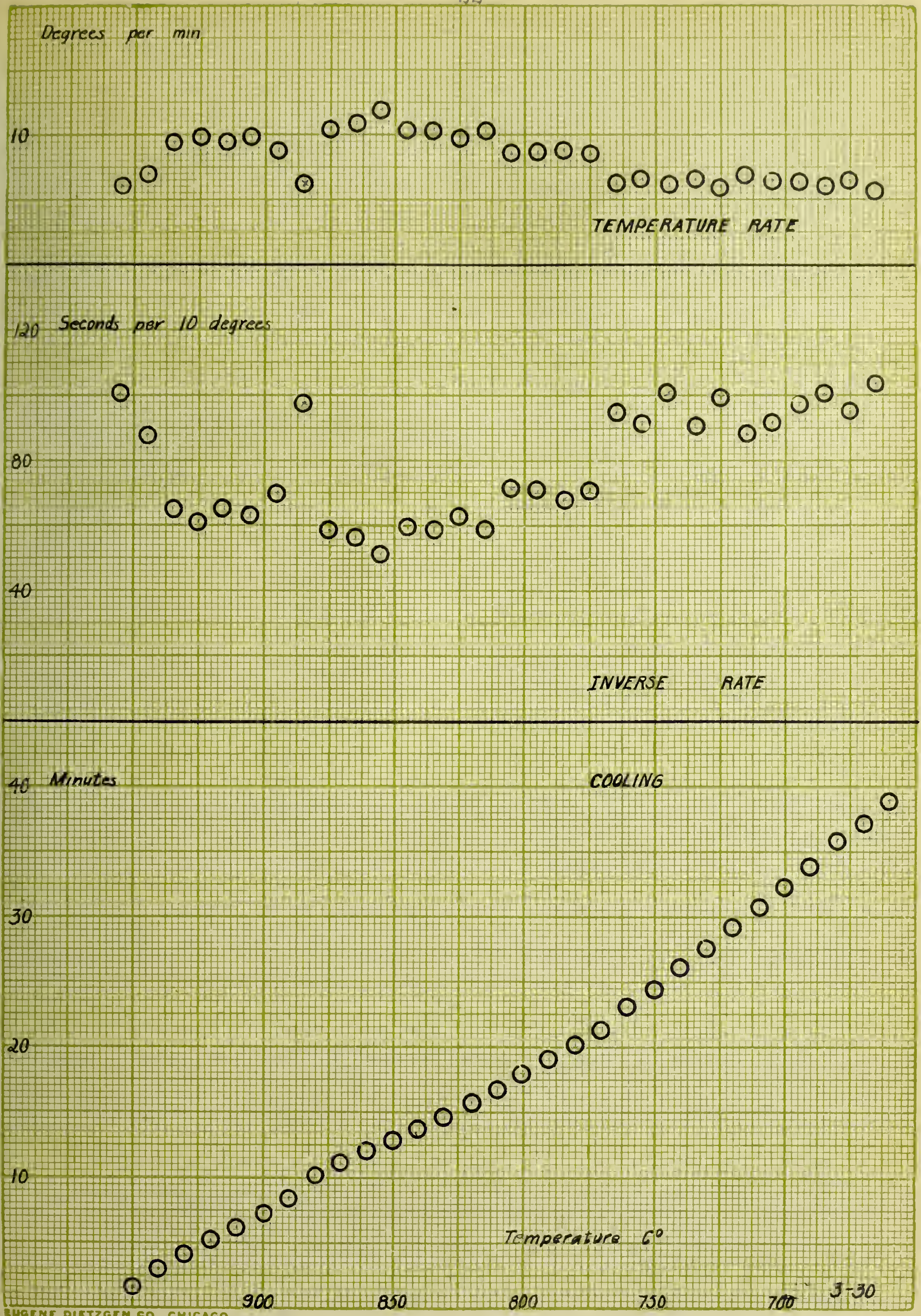

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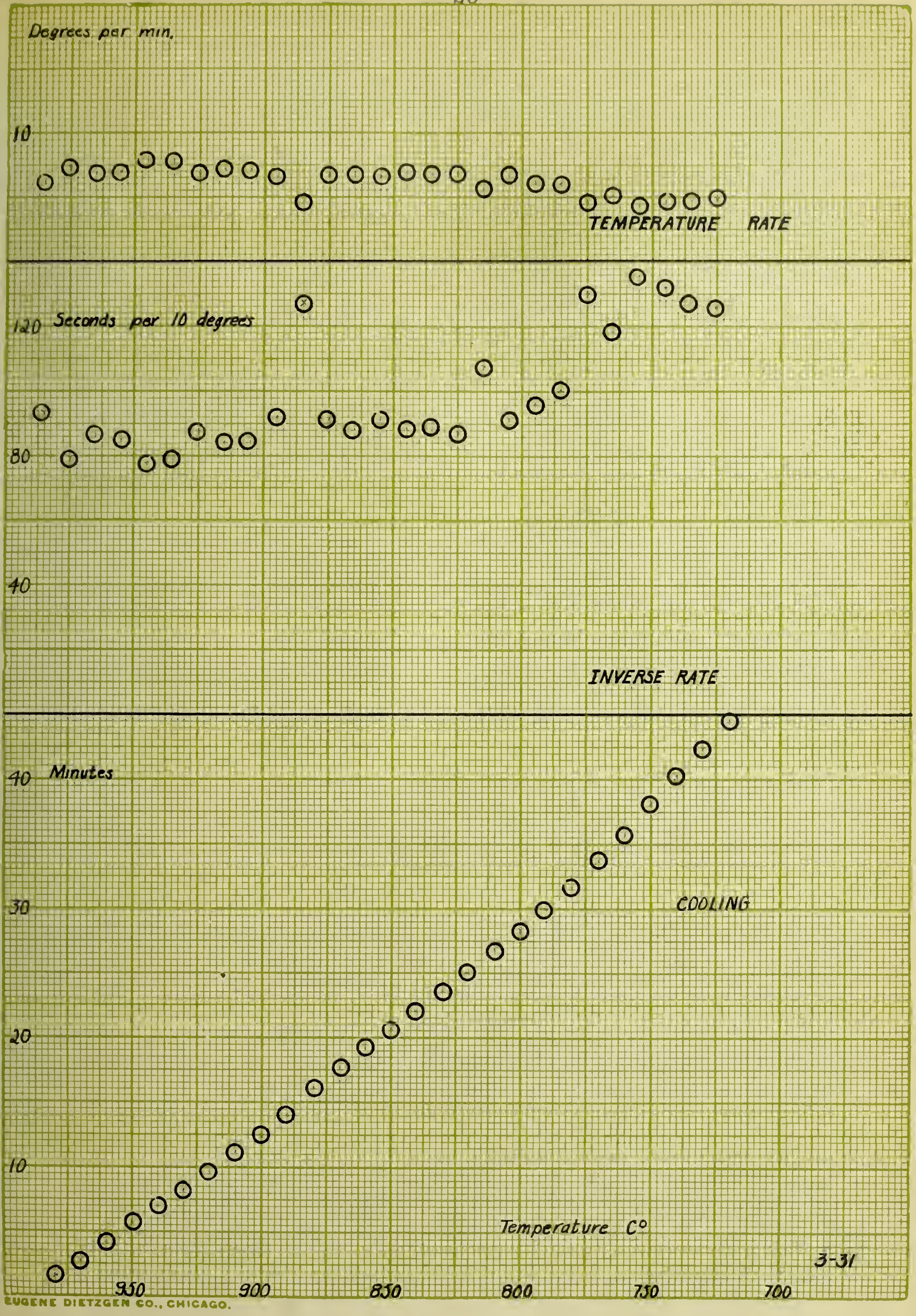
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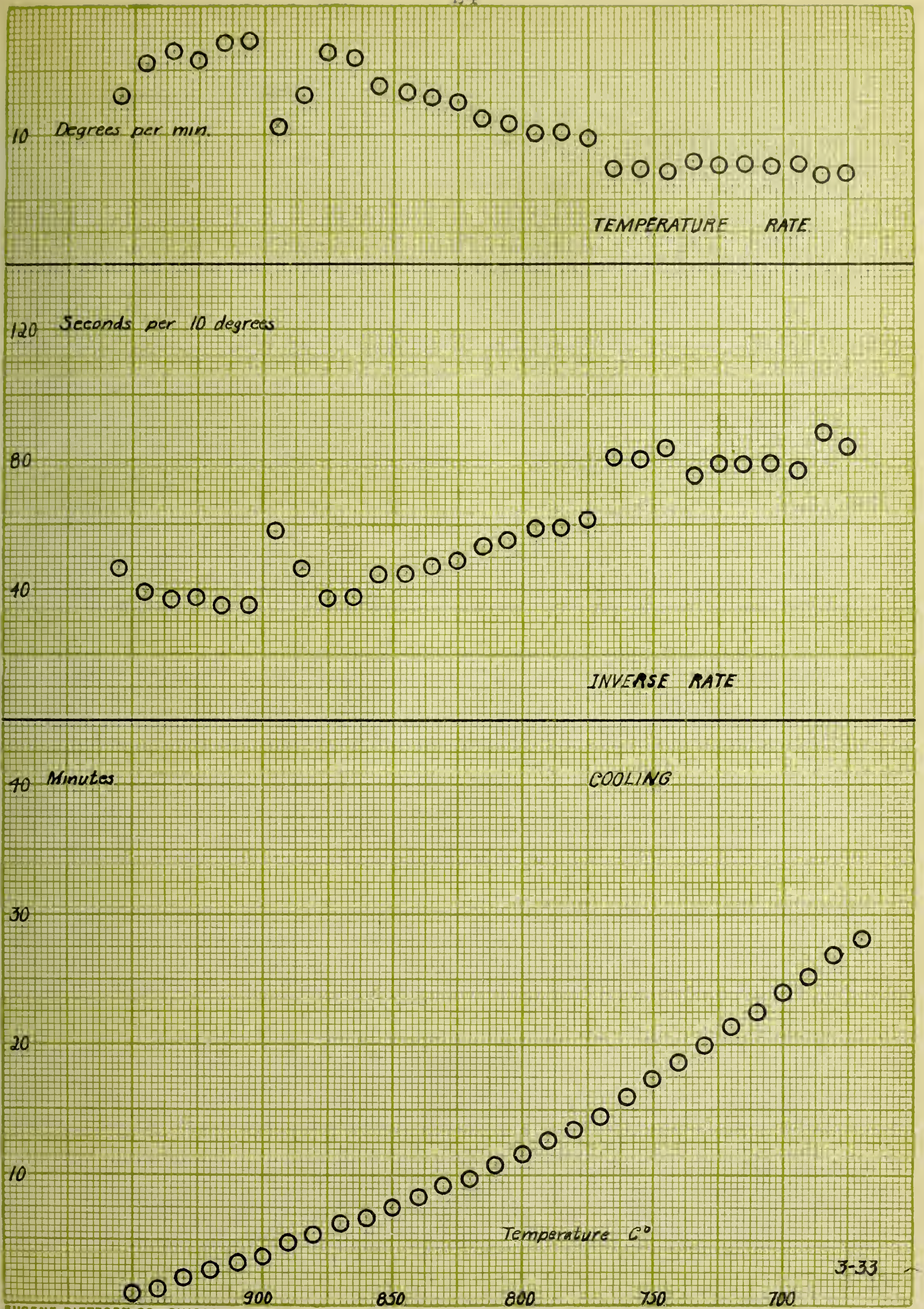

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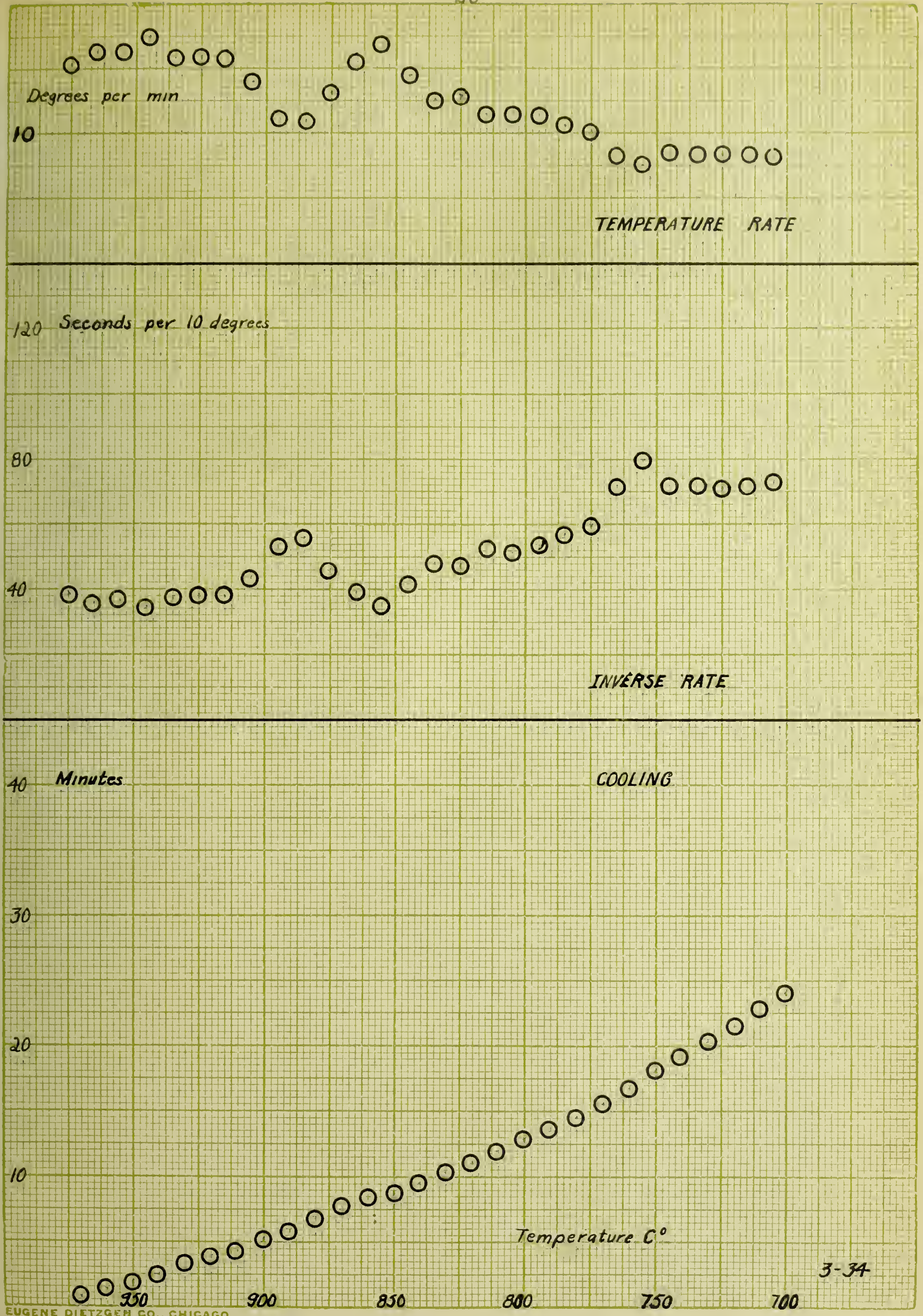

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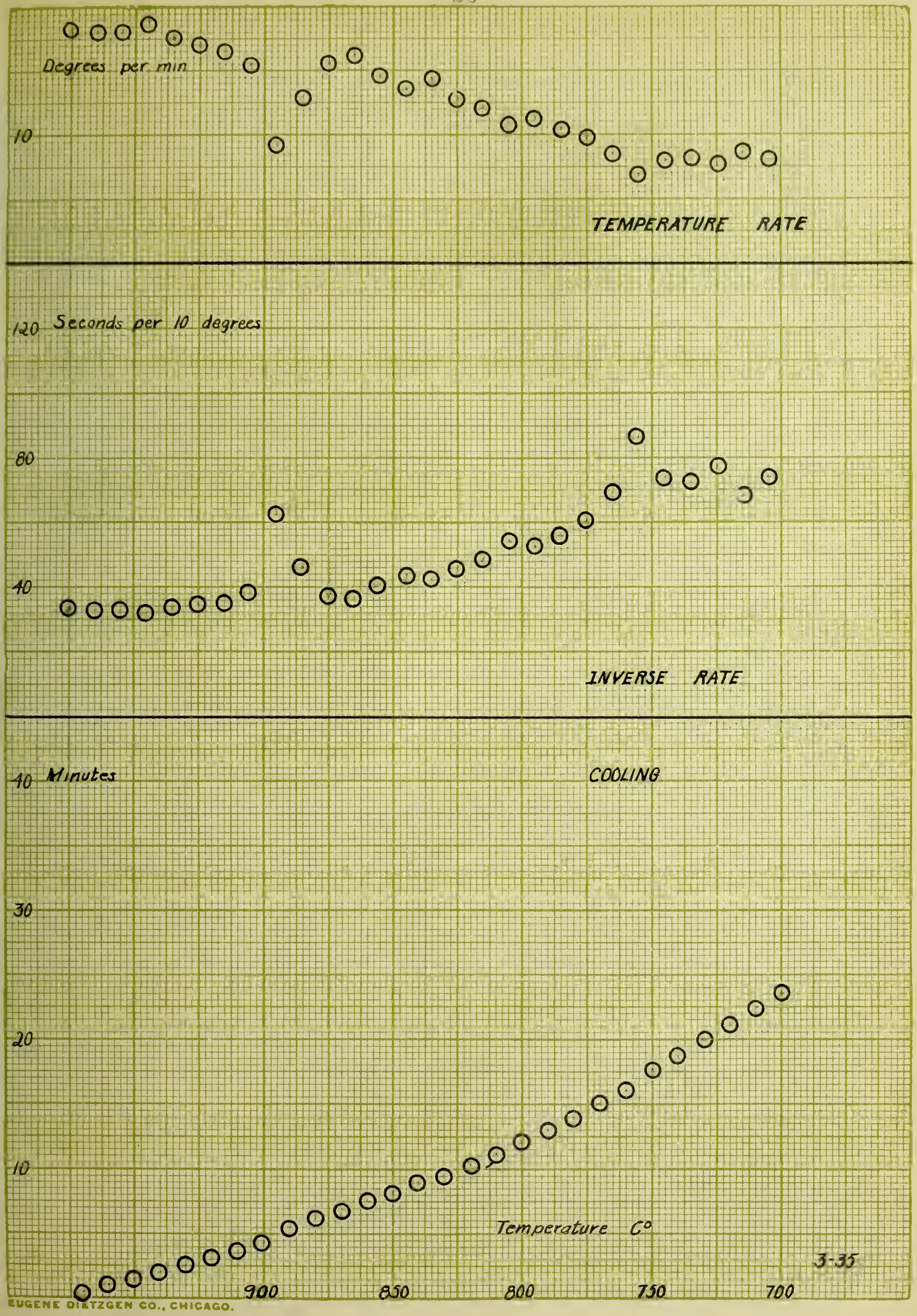
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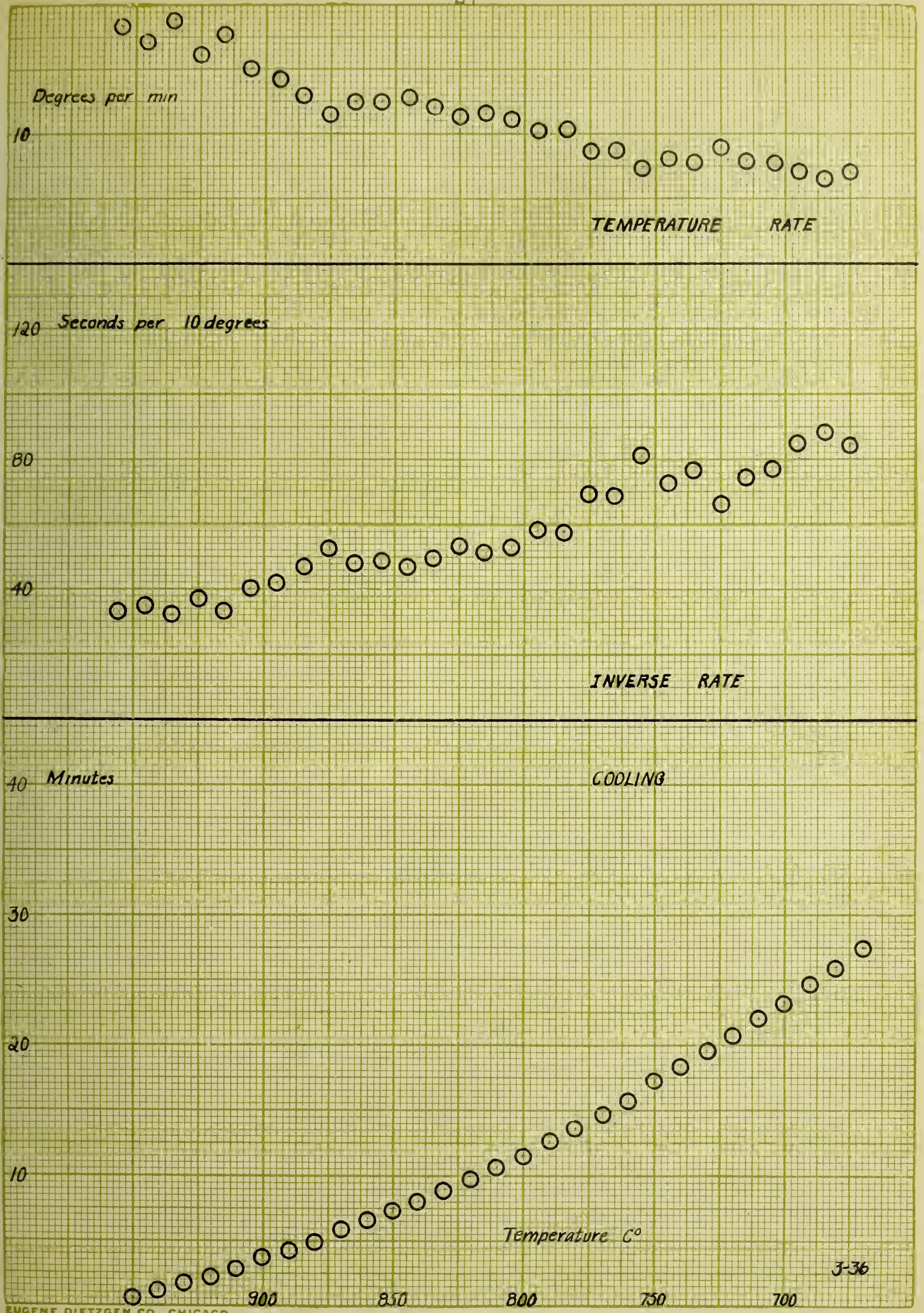

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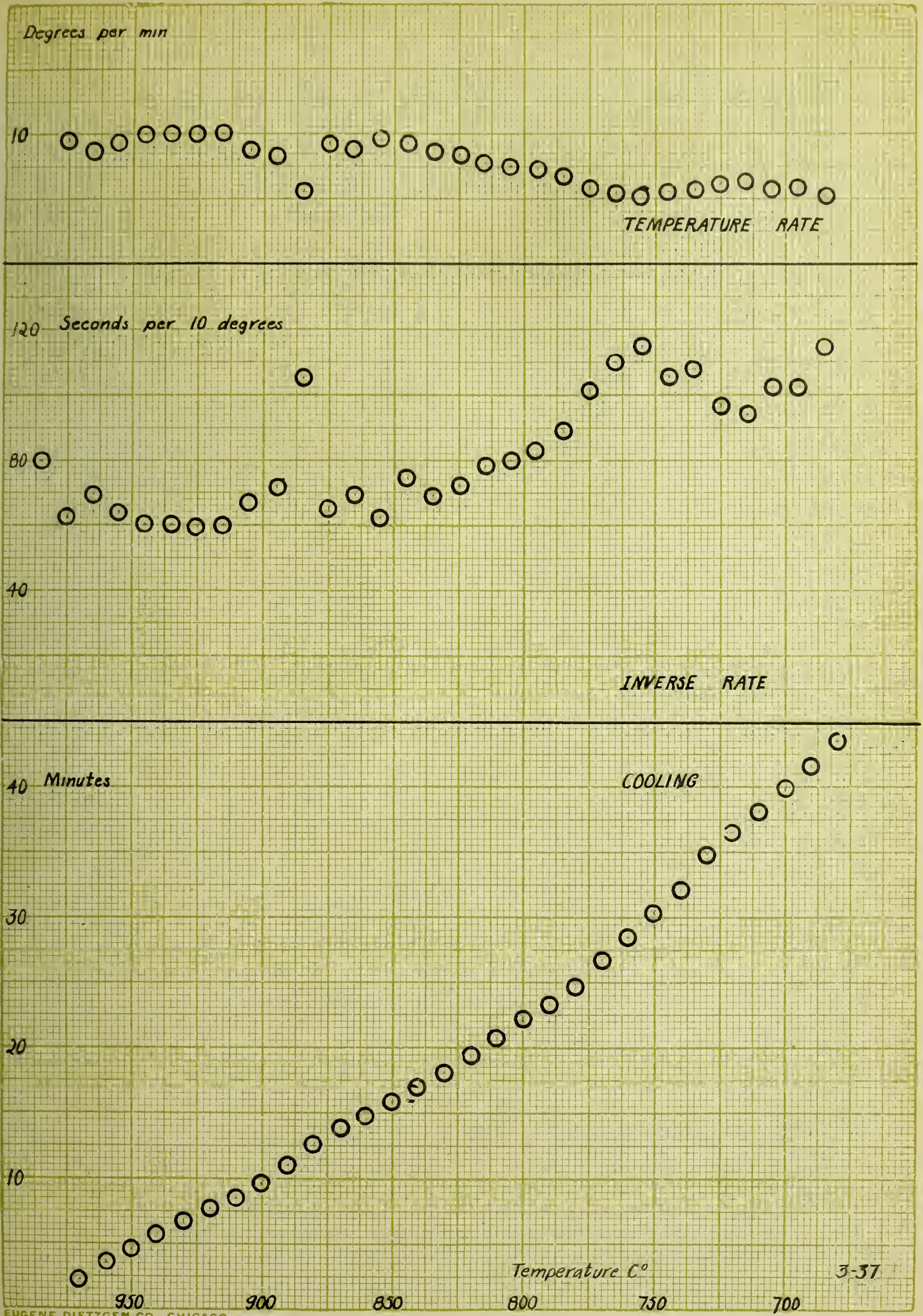

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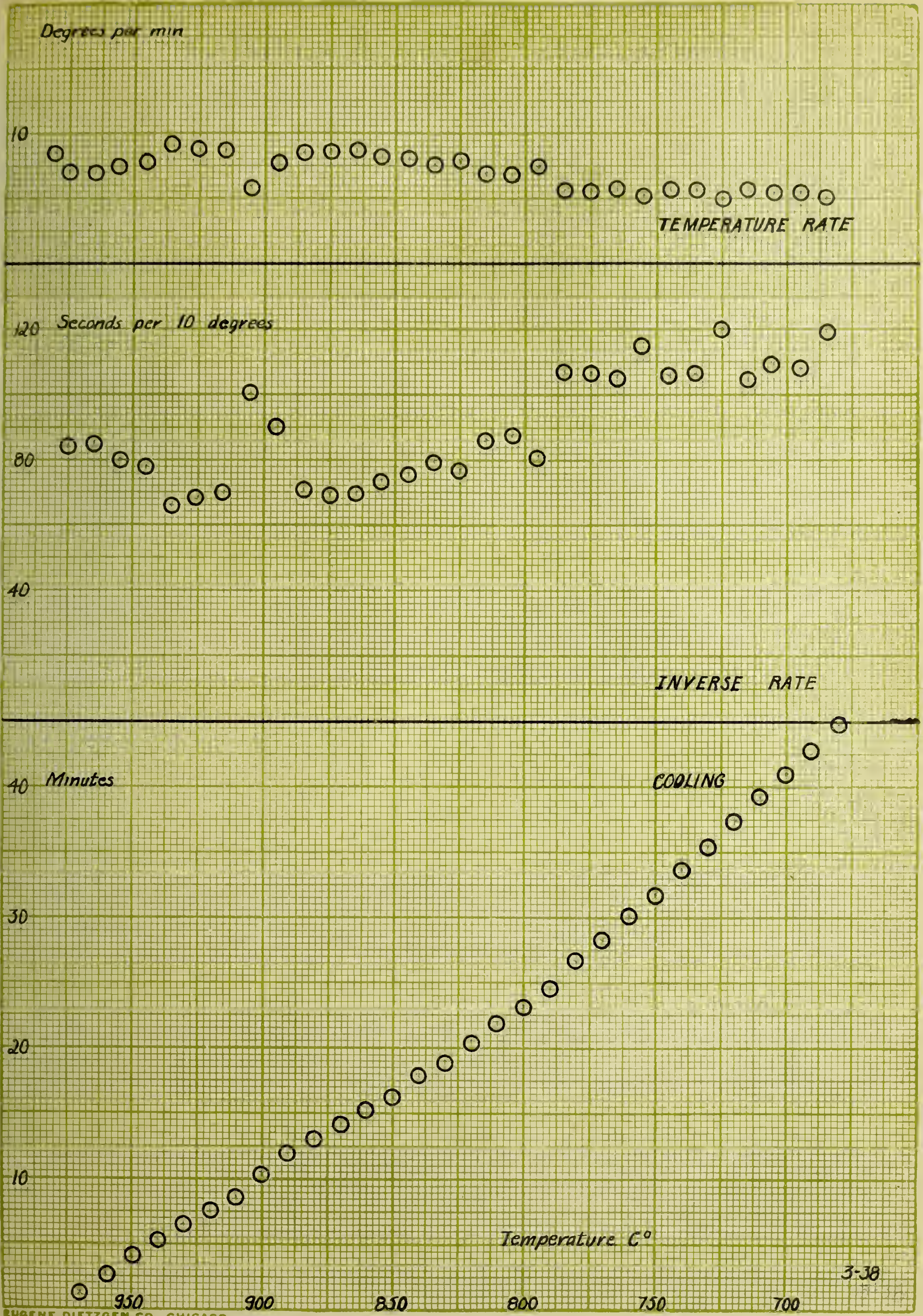

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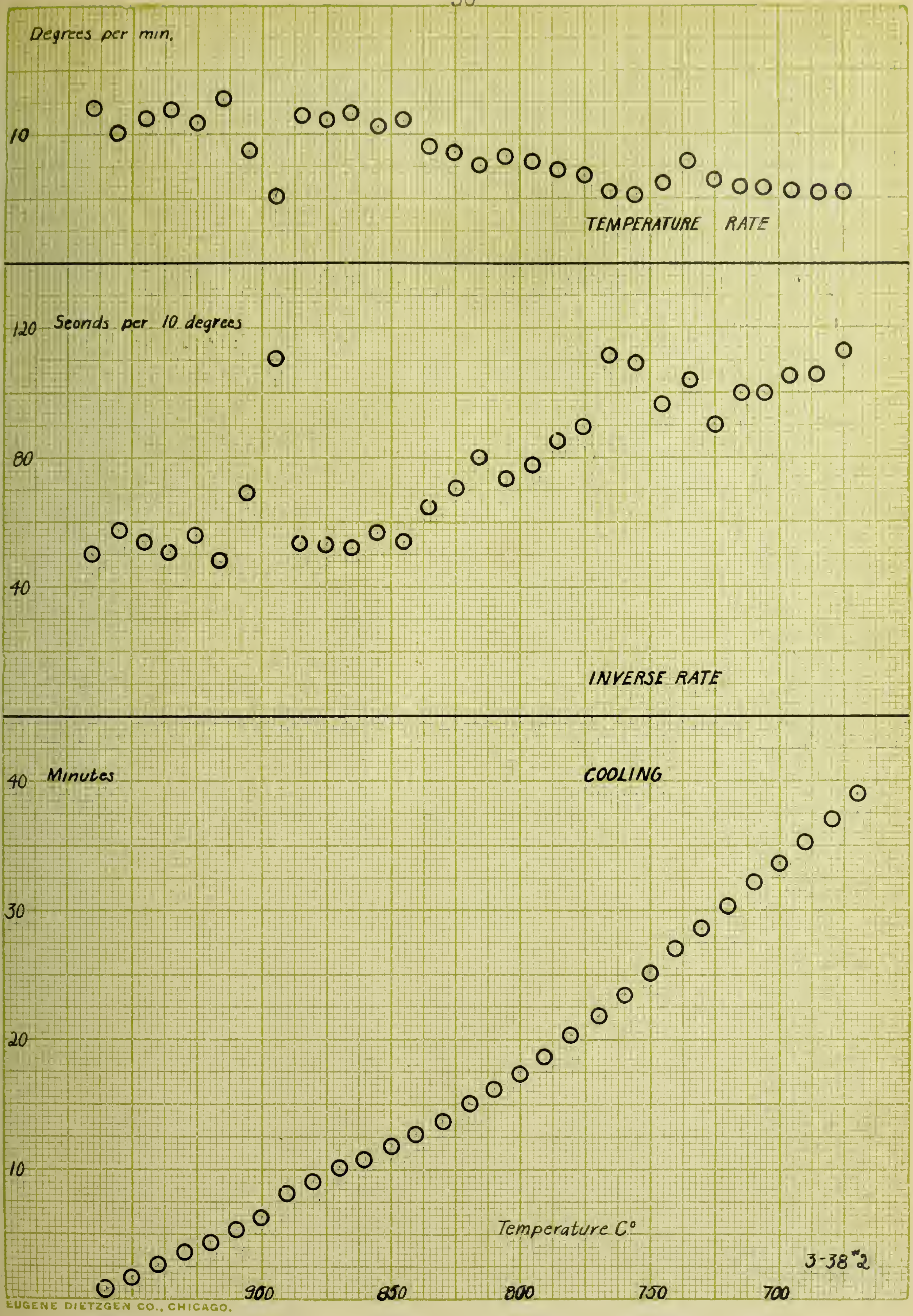
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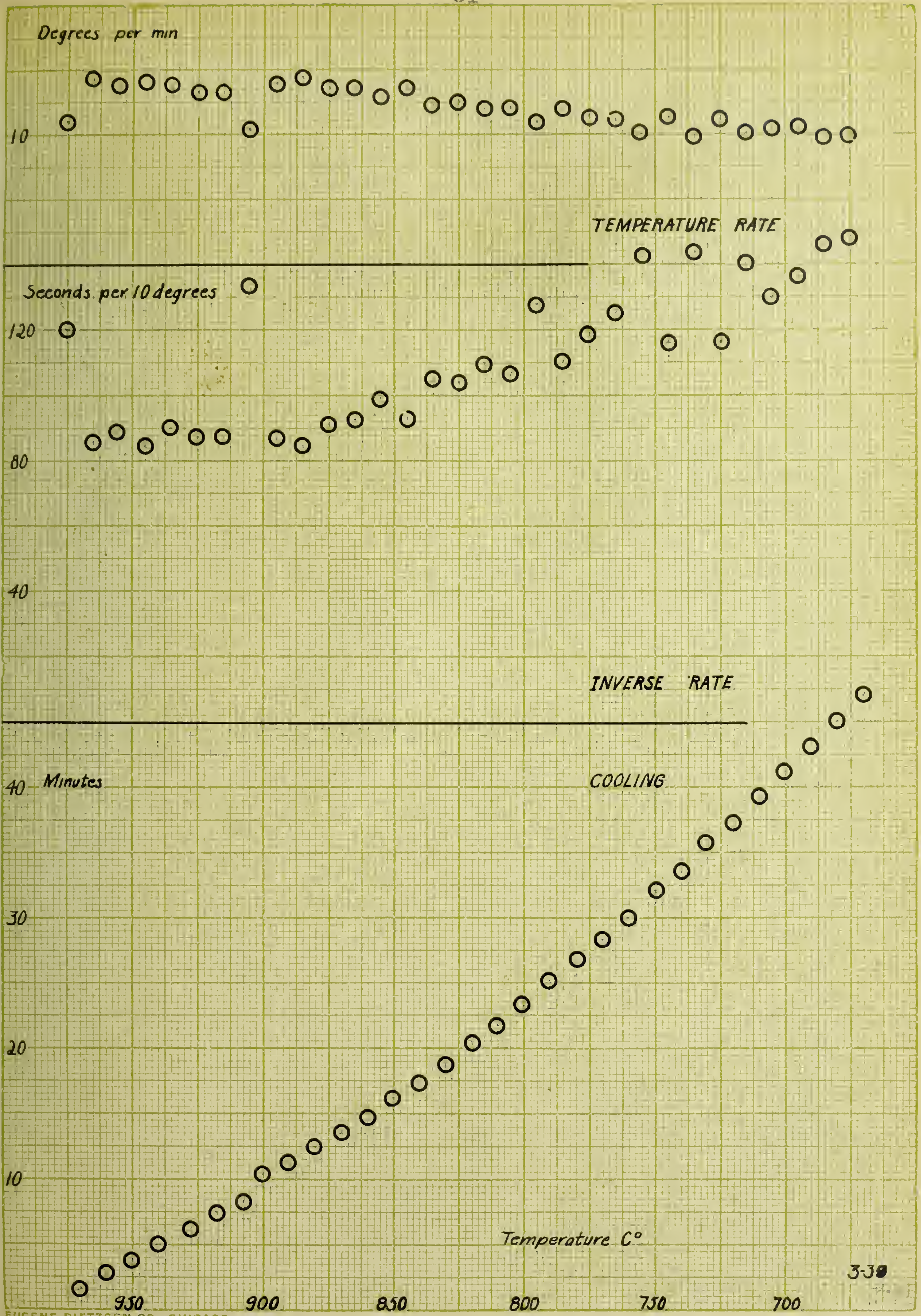

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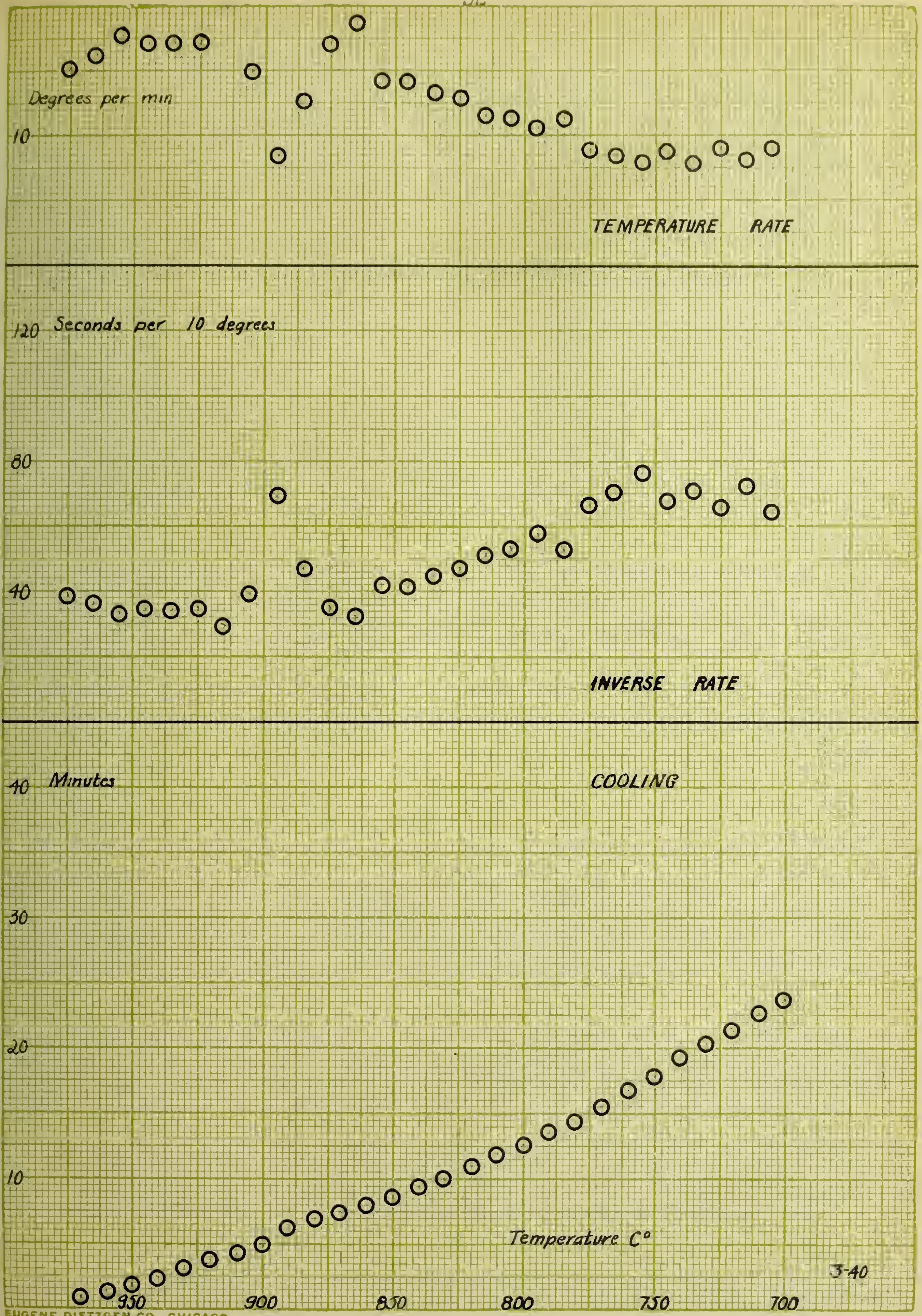

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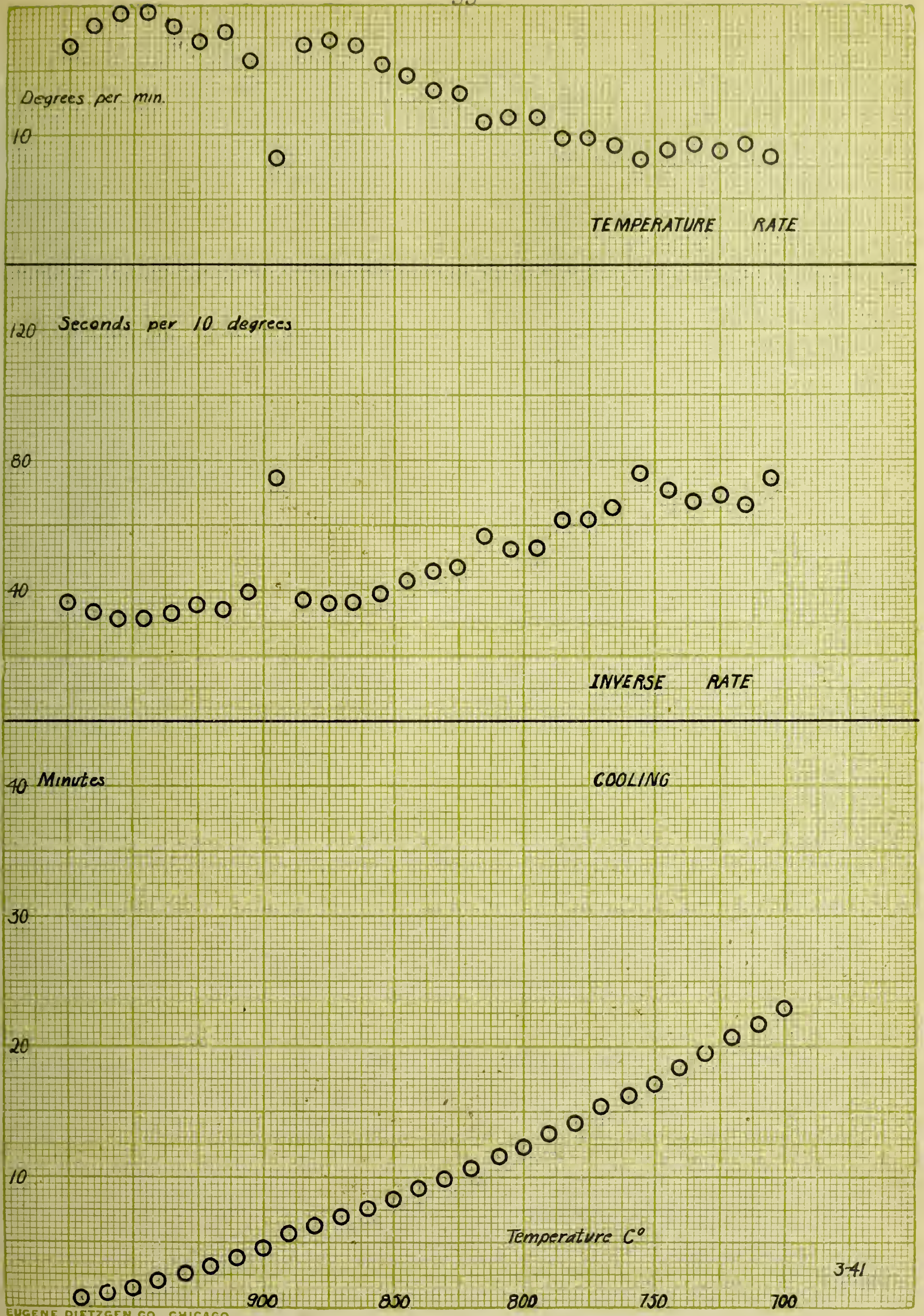

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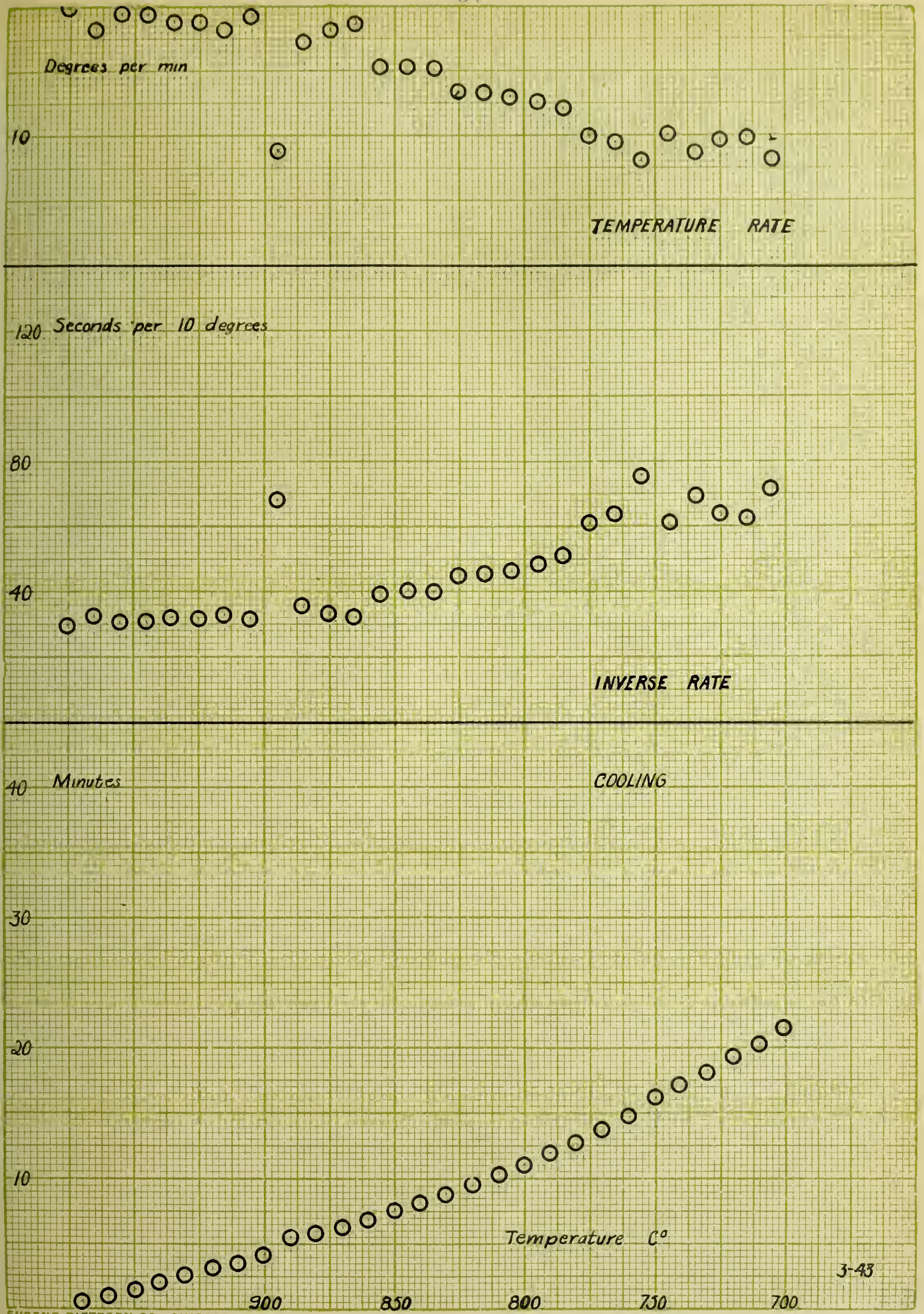

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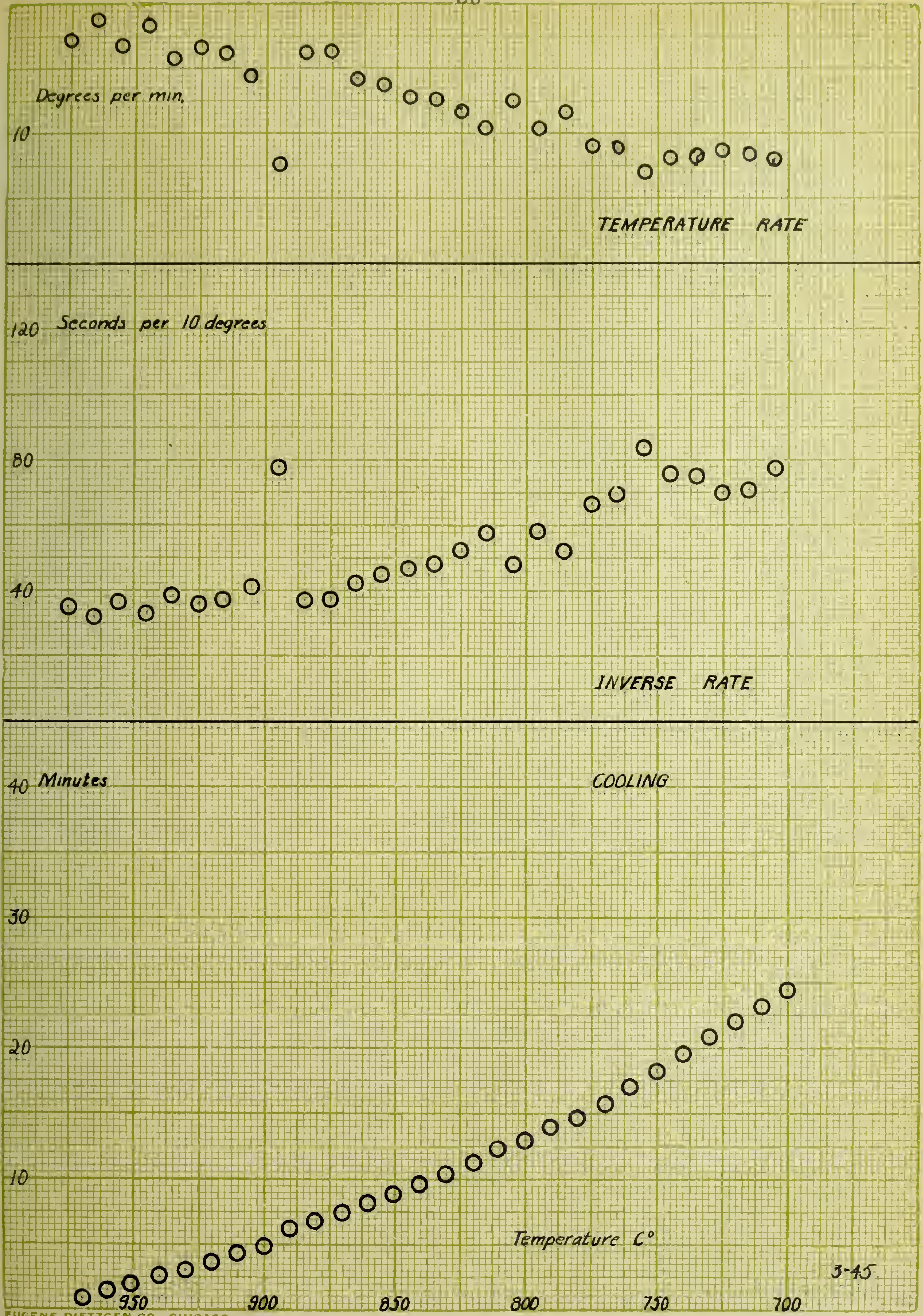

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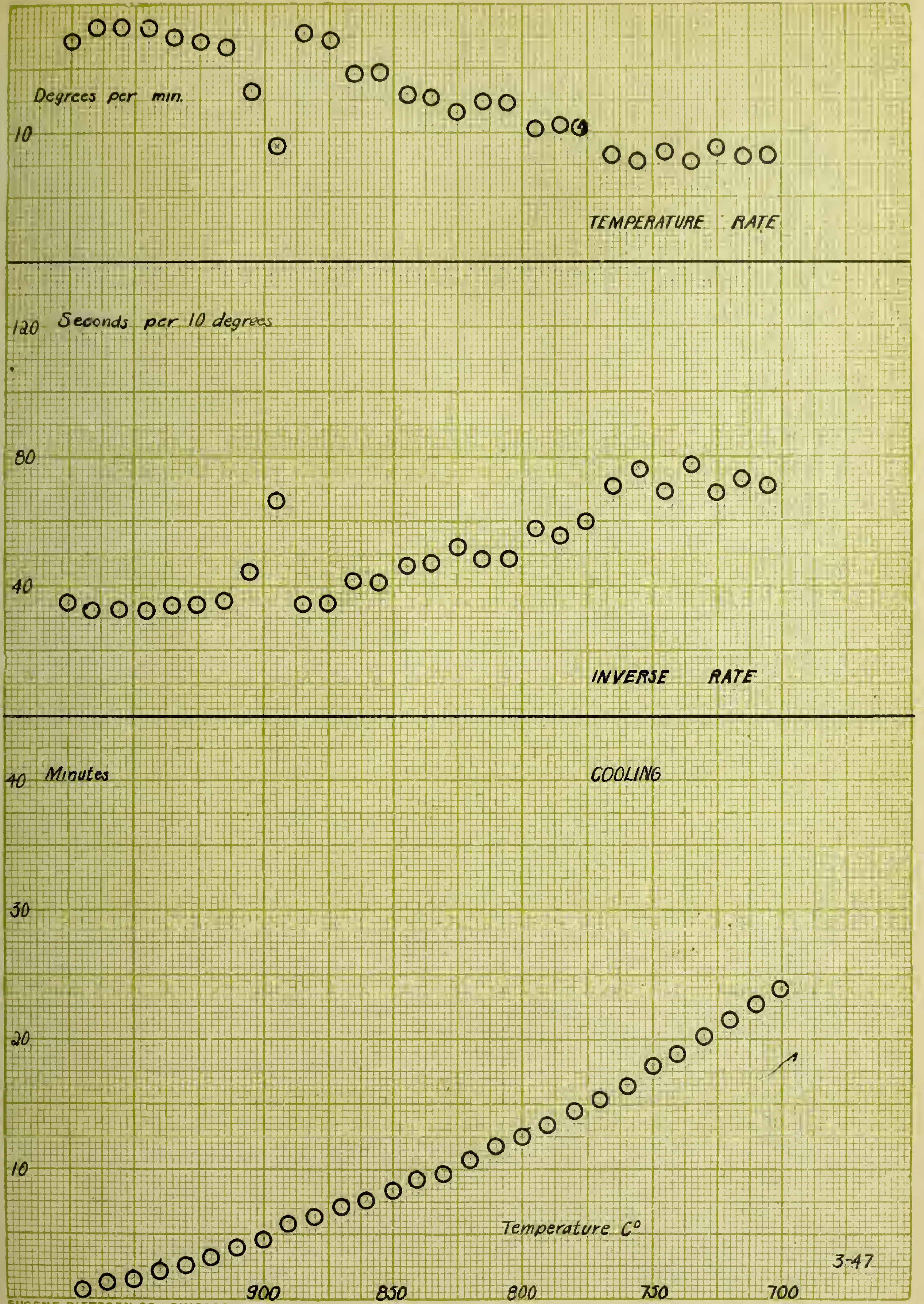

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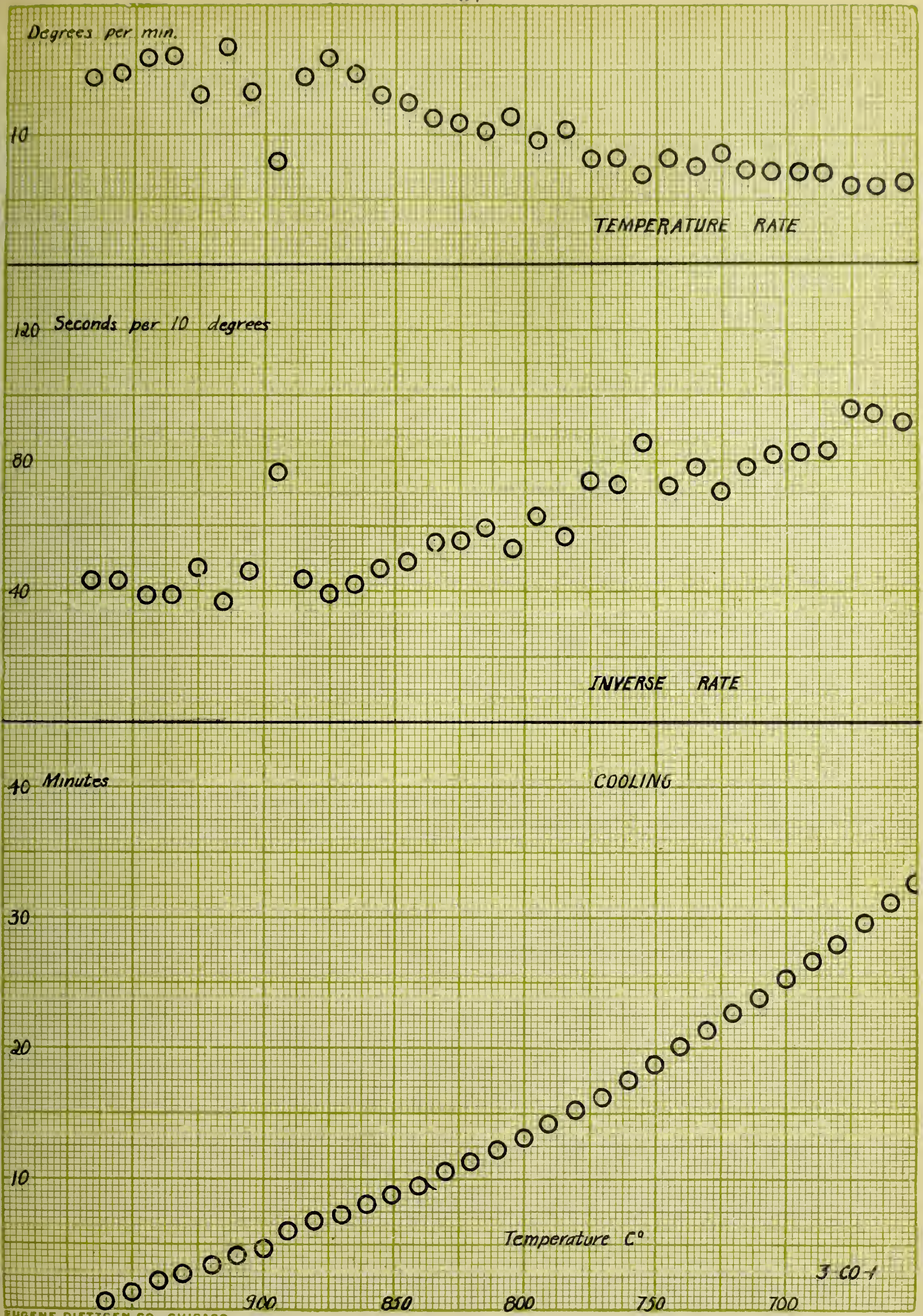

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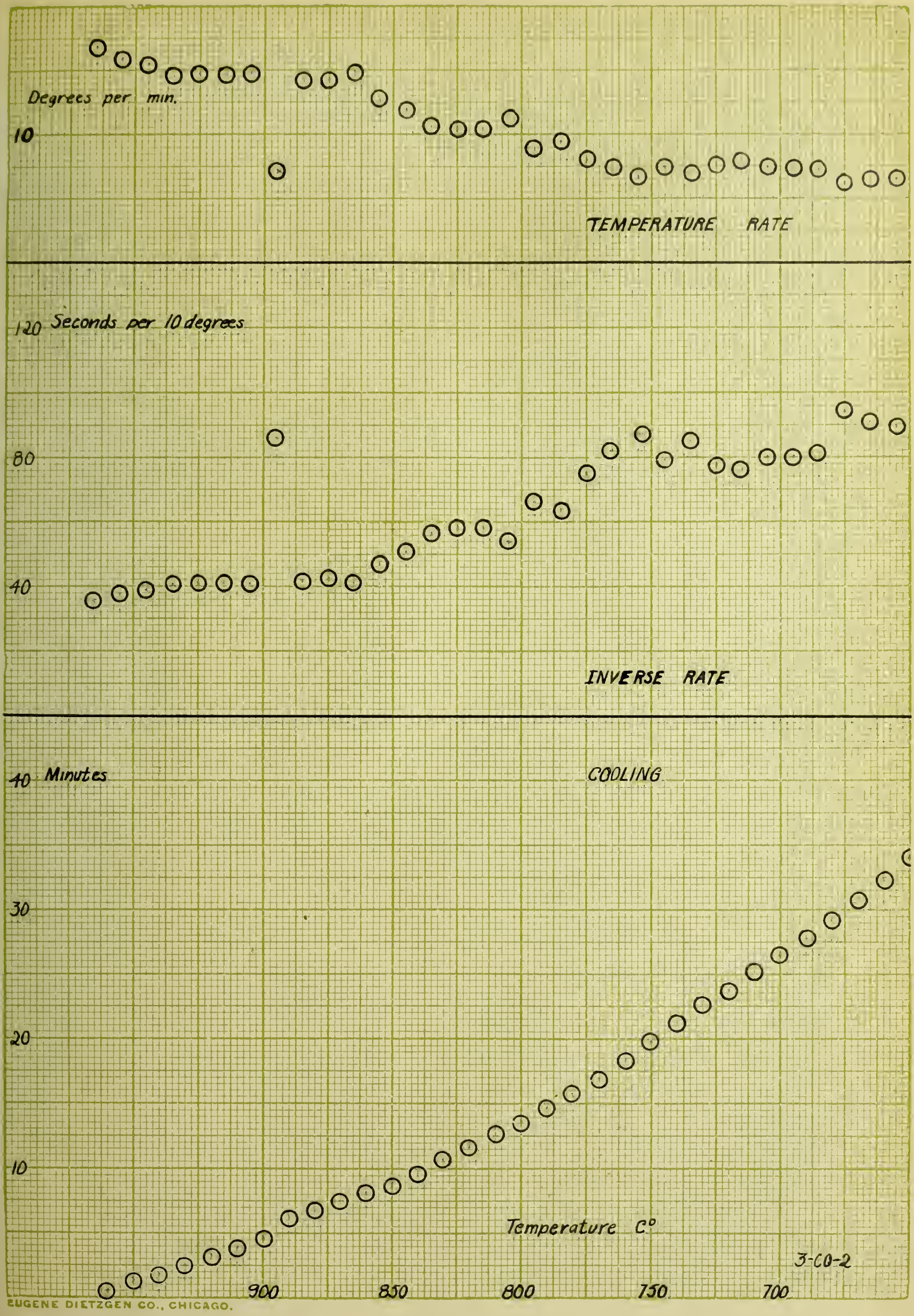
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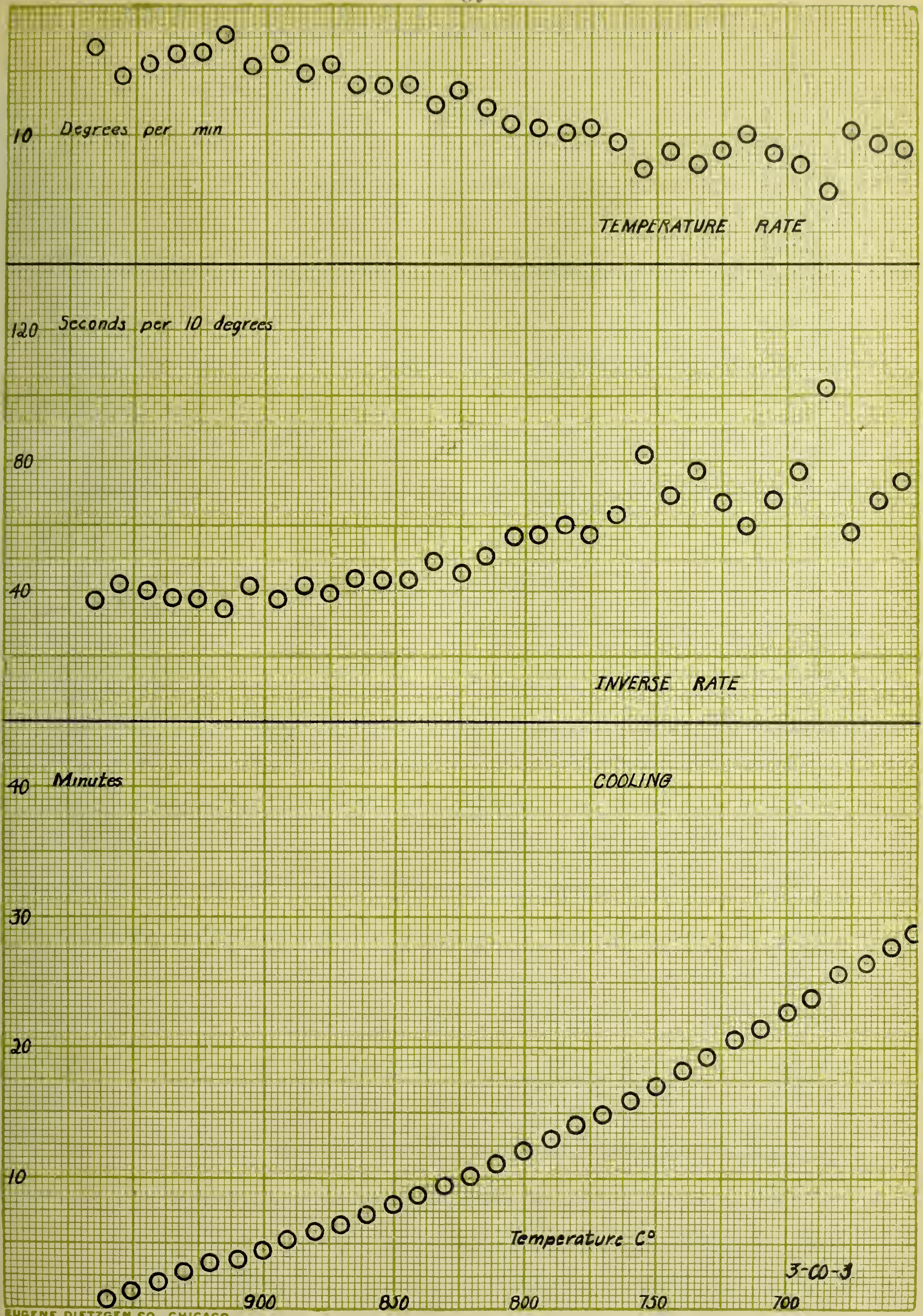

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III

THE RESULTS OF THE TENSILE TESTS

In the tables data are given for a series of tests ranging from the condition of forging to long annealing and very violent quenching. The method of obtaining these results is as follows.

Specimens tested as forged were precisely as obtained from the ingot after working under a steam hammer at forging heat and cooling in air. The lathe work is regarded as superficial in determining the properties of the iron.

The treatment for the specimens tabulated as annealed was invariable except for the time of cooling and the maximum temperature of heating. Preliminary to the heat treatment the specimens were placed in a large iron-pipe cartridge filled with crushed magnesite moistened with alcohol to drive out the air on heating and prevent scale formation. The cartridge and contents were then heated to the predetermined temperature in a plate type carbon resistance furnace having a maximum energy input of forty five kilowatts. Previous to beginning the heating a pyrometer tube was inserted through a hole in the cartridge until it penetrated the magnesite in contact with the specimens. This gave the means of determining the temperature.

In some of the annealing treatments the furnace was merely heated up to the required maximum and allowed to cool at its natural rate. This consumed from four to six hours and all specimens heated and cooled in this manner are designated as

furnace cooled. In case of the long annealings the furnace was heated as before, but the cooling was accomplished under a constantly decreasing energy input, the regulation being on the basis of the pyrometer readings following a definite rate.

In the case of the quenched specimens precisely the same mode of preliminary preparation was employed except that a smaller cartridge containing a lesser amount of magnesite was employed. After reaching the desired temperature the cartridge was removed from the furnace and its contents, including the magnesite quickly dumped in the cooling medium.

Specimens were tested in an Olsen machine of the beam balance type with a maximum capacity of five tons. Screw grips threading on the test pieces were employed so that very little slipping occured.

It was seen from the chemical analysis that the iron is peculiarly free from all impurities except carbon and that the carbon content suffered an increase with remelting in vacuo. It happens that the reaction of the molten charge with the rarefied furnace atmosphere is expressed by the equation,

$$Fe + CO = Fe\ O + C$$

which is reversible and whose direction of progression is determining by the pressure. With extremely low vacuum the reaction proceeds from right to left, while with the usual vacuum employed in melting the charge (2 or 3 mm. of Hg) it proceeds from left to right, and two undesirable constituents carbon and iron oxide are added to the charge. The variation in carbon content is chargeable to the change in the vacuum in melting the

different furnace charges and increases with increasing pressure. The iron obtained is of very high commercial purity indicating no trace of oxidation.

Specimen 3-37 evidently suffered some accident in the course of its preparation. It is unique among the electrolytic irons in point of its high carbon content.

RESULTS

The data obtained in these tests is presented in tables 3 to 7 . Table 3 is the general tabulation of the tests. It is impossible to plot curves for the reason that no system of coordinates is available for the graphs. Tables 4 to 7 are intended to take their places. In all of these tables under elongation, column (1) is the elongation at the yield point; column (2) is the elongation after failure.

Some of the conclusions to be derived from the data are partly negative; most of them are purely suggestive, although a few broad conclusions can be drawn which are not intrinsically applicable to electrolytic iron.

As in the case of almost all mechanical tests not enough specimens were tested.To settle definitely many of the points involved a much wider series must be completed, but the tests are believed to be valuable in suggesting the following points.

With the forged specimens the values indicate very great confusion. This was to be expected from the analogous behaviour of other alloys and was the reason for testing the specimens in this form. The higher strength is due to mechanical

work while passing through the range A_3, and seems to be independent of the carbon content.

With the annealed specimens it may be said that very little grain refining is secured, and that the result is merely the elimination of the cooling strains. The net return of the annealing is uniformity. The time of the annealing is not essential provided it is sufficiently long to accomplish the desired end. The refinement of following a logarithmic or straight line curve is entirely superfluous. Twenty four hours is probably sufficient to remove all harmful cooling strain.

In the quenching experiments the same criticism as with the annealing may apply. Because of the fact that almost no grain refining was secured, the end of the quenching process was only partially obtained. It is probable that some grain refining was secured because of the higher temperature of quenching; it is also possible that some of the higher allotropic modifications are retaining in the cold state. Either of these assumptions will account for the slight increase in strength, or it may be ascribed to the fact that the rapid heating did not entirely destroy the advantageous grain structure of the forged specimen.

Carbon variation even with the small amount of the element involved produces a change in the strength of all but the forged specimens in which it is very likely obliterated by other influences. It appears to affect the yield point much more markedly than the ultimate.

In some of the tests the yield point could not be detected. It appears that it occured at such a low value that it remained undiscovered in the confusion of getting the test machine under way.

In the rupture of the annealed sections it was noted that the section was greatly reduced, irregular in cross section, seldom round and usually rectangular, while with the forged and quenched sections the fracture was invariable circular. At the moment of rupture the annealed metal was so plastic that it was sometimes difficult to join the ends together to measure elongation. The fracture of the other specimens showed a crystalline break.

The elongation of the annealed specimens was nearly uniform over the entire length until the moment of necking. The progress to destruction was then very rapid. The necking of the forged and quenched specimens was relatively much more localized.

It is probable that the quenched test pieces were under a very severe cooling strain. Punch marks in several of them showed that in the quenching they had been twisted through an angle of approximately thirty circular degrees.

One specimen was quenched in liquid air. It showed a much more uniform elongation than any of the other quenched test pieces either from more uniform or less violent treatment. For large masses iced brine is believed to be a much more effective cooling medium than liquid air despite the latter's lower temperature. However, for small specimens it is believed that in

quenching from slightly above the A_3 point the allotropic transformation of the iron is greatly retarded due to the initially greater cooling effect. Liquid air tends to volatilize and surround the specimen with a then film of air so that the effect of its low temperature is partly lost.

It is believed that the results given herein are unusual in showing lower strength and greater ducility than is usually associated with iron. It is easy to produce iron of equally low carbon content with the ones shown here. It is almost impossible by any other method to secure a product so free from occluded gases and oxide or the presence of a deoxidising agent and this is regarded as the principal reasons for the oddity of the results obtained.

General Log Sheet

Table 3

imen	%	As Forged A					Annealed from 900 to 200 °C in 12 hrs acc to Straight Line B					Annealed from 900°-200°C in 12 hrs acc to Log Curve C					Quenched from 1000°C in Brine D					Remarks
	C	Yield Pt lbs/sq in	Ult Strenght lbs/sq in	Elong 1	2	Red of Area %	Yield Pt lbs/sq in	Ult Strenght lbs/sq in	Elong 1	2	Red. of Area %	Yield Pt lbs/sq in	Ult Strenght lbs/sq in	Elong 1	2	Red of Area %	Yield Pt lbs/sq in	Ult. Str. lbs/sq in	Elong 1	2	Red of Area %	
30		36600	44800		41	87.7																
31		69100	70500		20	82.8	20700	35500	30	60	89.5											
33		64000	68000		22	81.5												40500	21	53	89.5	
34													34300		47	61.1		55500	14	32	83.1	
15												14400	34500		54	87.3		44900	22	44	86.0	
16												20100	37500		52	77.0						
7																	41250	64600	15	37	73.7	
8													35150		56	85.5	37400	45500	17	42	86.1	
39		35800	44700		39	80.4						16400	36100		61	80.9						Column C Annealed in 48 hrs
40		39600	43500		38	82.3		41600		42	85.7											
41		62200	63500		26	83.8		40200	18	53	85.1											
43							18750	35800	31	59	84.5											
45		51400	55500		32	85.8	15000	39400	27	40	88.0											
47							17400	36100	31	57	91.3						30200	40000	20	47	80.8	Column D Quenched in Liquid Air
48		45600	51800	12	36	86.4						15300	35800	23	42	73.2						Column Annealed in 24 hrs
49		41400	48700	12	44	76.2						11100	34900	23	32	75.7						900 - 200 °C
50		38200		12.5	35							19300	35800	28	45	86.5						acc to Straight Line
0-1							21000	57100	27	41	72.5						33200	53000	20	65	86.2	% Carbon Added .05
0-2							19400	38900	29	60	73.5						67700	76900	17	25	79.7	" " " .10
0-3							22100	51000	24	50	64.5											" " " .50

AS FORGED

Table 4

Specimen Number	Maxima					Minima					Average					Remarks
	Yield Point lbs/sq in	Ultim Strength lbs/sq in	Elong 1	Elong 2	Reduct of Area o/o	Yield Point lbs/sq in	Ultim Strength lbs/sq in	Elong 1	Elong 2	Reduct of Area o/o	Yield Point lbs/sq in	Ultim Strength lbs/sq in	Elong 1	Elong 2	Reduct of Area o/o	
3-30					87.7											
3-31	69100	70500														
3-33									20							
3-34																
3-35																
3-36																
3-37																
3-38																
3-39						35800										
3-40							43500									
3-41															—	
3-43																
3-45												+		—		
3-47																
3-48								12			—		∸			
3-49				44				12		76.2			—			
3-50			12.5													
Average											48390	54800	12.2	33.3	83.9	
3-CO-1																
3-CO-2																
3-CO-3																
Average																

ANNEALED 900 - 200°C 12 HRS STRAIGHT LINE Table 5

pecimen	Maxima					Minima					Average					
umber	Yield Point lbs/sq in	Ultim. Strength lbs/sq in	Elong 1	2	Reduct of Area o/o	Yield Point lbs/sq in	Ultim Strength lbs/sq in	Elong 1	2	Reduct of Area o/o	Yield Point lbs/sq in	Ultim Strength lbs/sq in	Elong 1	2	Reduct of Area o/o	Remarks
3-30																
3-31	20700			60			35500									
3-33																
3-34																
3-35																
3-36																
3-37																
3-38																
3-39																
3-40		41600														
3-41								18						+		
3-43			31							84.5						
3-45						15000			40			+	−		+	
3-47			31		91.3						−	+				
3-48																
3-49																
verage											17960	38100	27.4	51.8	87.4	
3-CO-1		51100							41							
3-CO-2			29	60	73.5	19400	38900									
3-CO-3	22100							24		64.5						
verage											17500	46800	26.6	50.3	70.1	

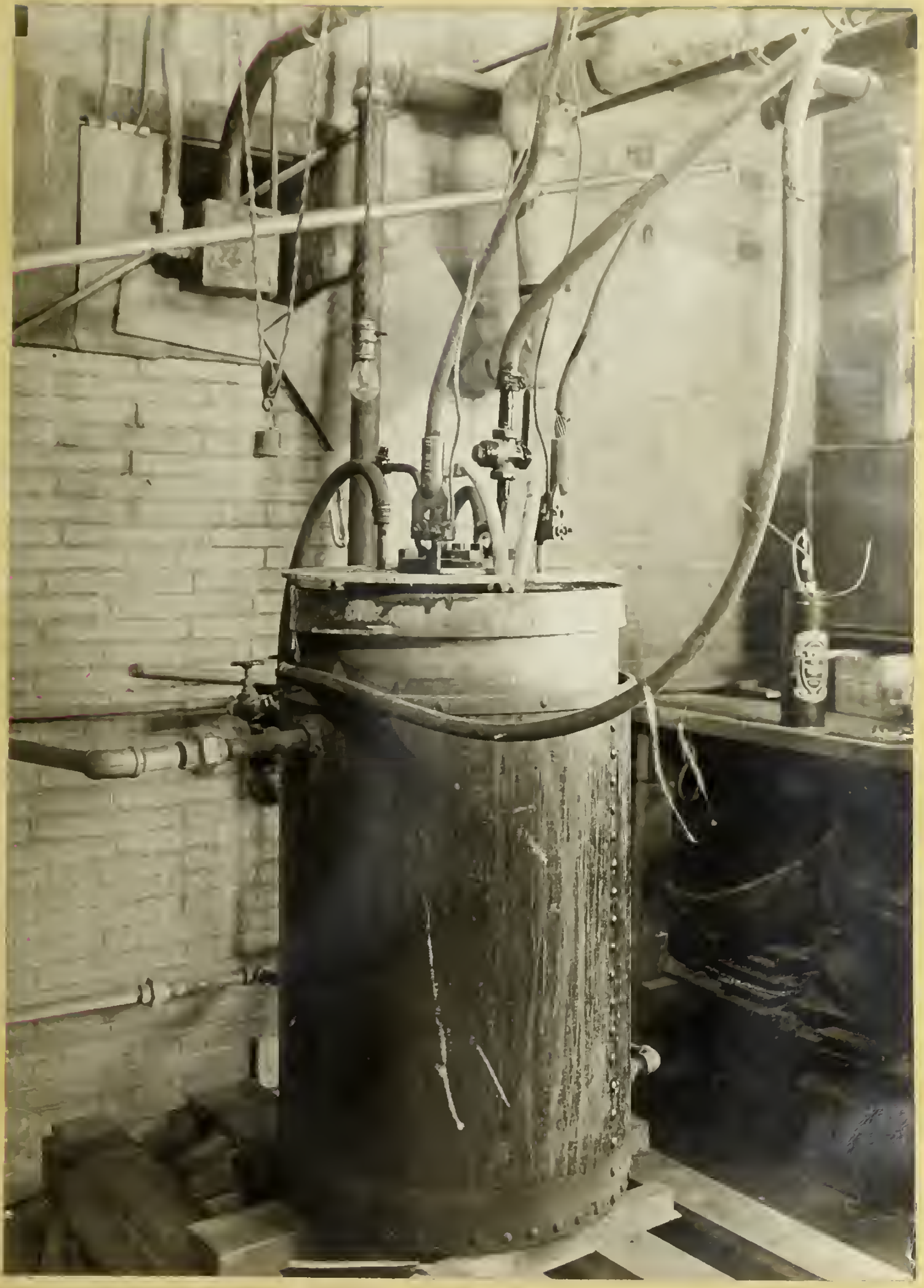
ANNEALED 900 - 200°C 24 HRS LOG CURVE STRAIGHT LINE Table 6

Specimen	Maxima					Minima					Average					
Number	Yield Point lbs/sqin	Ultim Strength lbs/sqin	Elong 1	Elong 2	Reduct of Area o/o	Yield Point lbs/sqin	Ultim Strength lbs/sq in	Elong 1	Elong 2	Reduct of Area o/o	Yield Point lbs/sqin	Ultim Strength lbs/sqin	Elong 1	Elong 2	Reduct of Area o/o	Remarks
3-30																
3-31																
3-33																
3-34							34300			61.1				—		
3-35					87.3											
3-36	20100	37500													—	
3-37																
3-38																
3-39				61							+					
3-40																
3-41																
3-43																
3-45																
3-47																
3-48								23					—			
3-49						11100		23	32				—			
3-50			28									+				
Average											16100	35500	24.7	48.6	78.4	
3-CO-1																
3-CO-2																
3-CO-3																
Average																

QUENCHED 1000-(-21) °C ICED BRINE

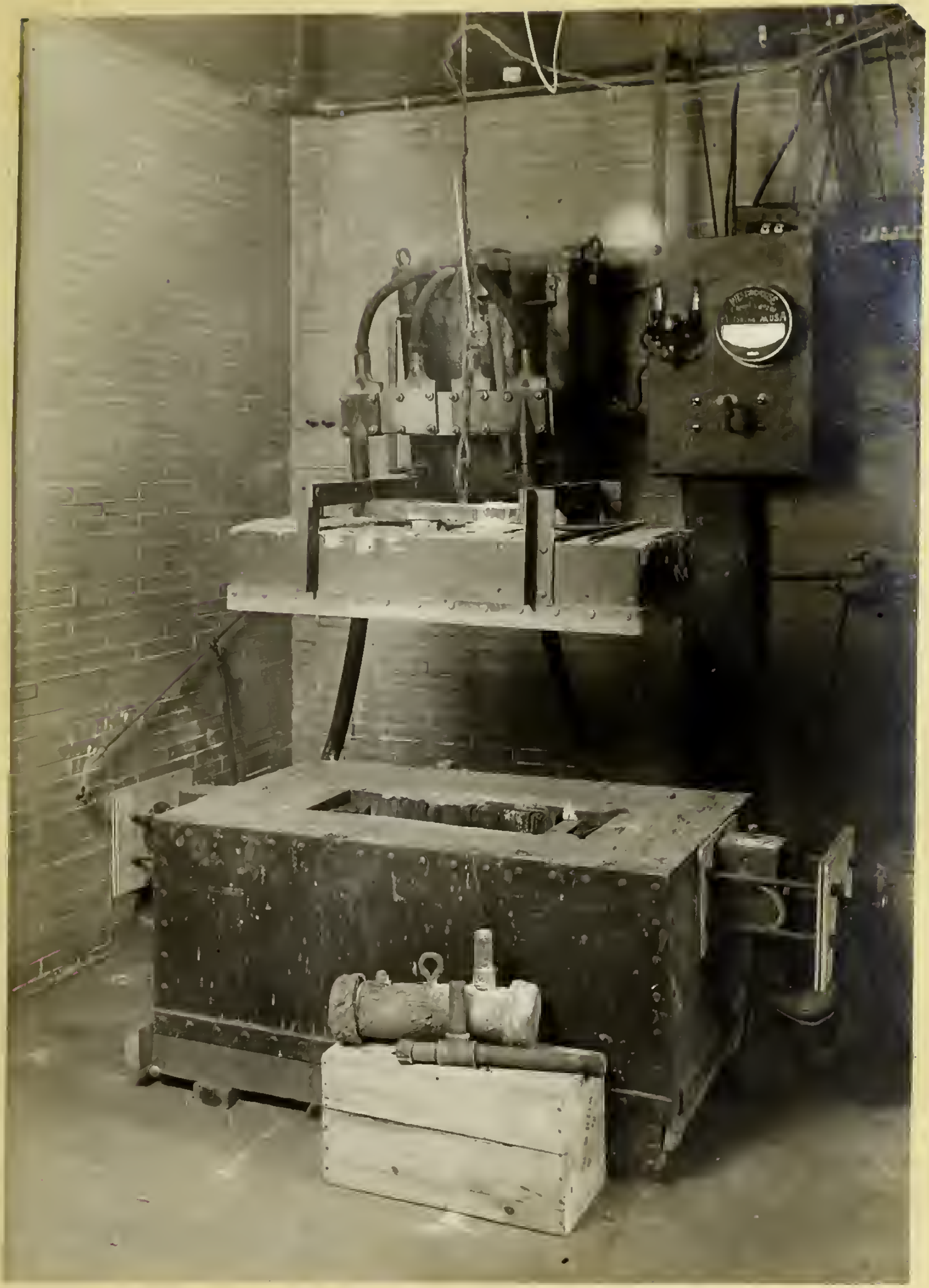
Table 7

pecimen	Maxima					Minima					Average					
umber	Yield Point lbs/sq in	Ultimate lbs/sq in	Elong 1	2	Reduct of Area o/o	Yield Point lbs/sq in	Ultimate lbs/sq in	Elong 1	2	Reduct of Area o/o	Yield Point lbs/sq in	Ultimate lbs/sq in	Elong 1	2	Reduct of Area o/o	Remarks
3-30																
3-31																
3-33			24	53			40500									
3-34								14	32			+			+	
3-35																
3-36																
3-37	41250	64600								73.7						
3-38					86.1								–	–		
3-39						37400										
3-40																
3-41																
3-43																
3-45																
3-47																
3-48																
3-49																
3-50																
Average											36280	50170	18.6	42.5	82.4	
3-CO-1																
3-CO-2																
3-CO-3																
verage											50450	64950	10.3	45	82.9	Two Specimens only



VACUUM FURNACE
Fig 1

[illegible]
[illegible]
UNIVERSITY OF [illegible]



RESISTANCE FURNACE
Fig 2

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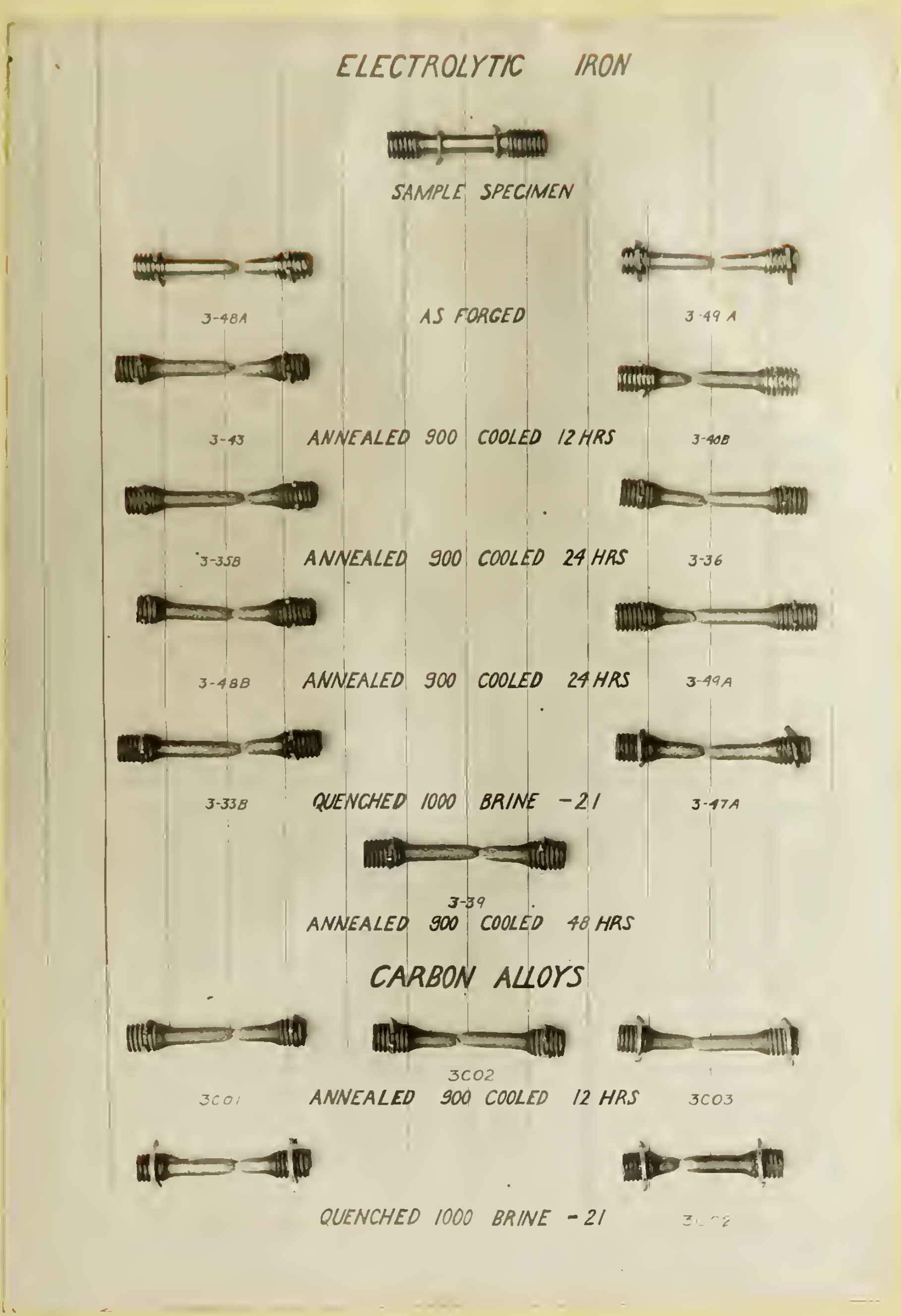
ELECTROLYTIC IRON
SAMPLE SPECIMEN
3-48A
AS FORGED
3-49 A
3-43
ANNEALED 900 COOLED 12 HRS
3-40B
3-35B
ANNEALED 900 COOLED 24 HRS
3-36
3-48B
ANNEALED 900 COOLED 24 HRS
3-49A
3-33B
QUENCHED 1000 BRINE -21
3-47A
3-39
ANNEALED 900 COOLED 48 HRS
CARBON ALLOYS
3C02
3C01
ANNEALED 900 COOLED 12 HRS
3C03
QUENCHED 1000 BRINE -21

SPECIMENS OF TENSILE TESTS
Fig 3

TEST SPECIMEN

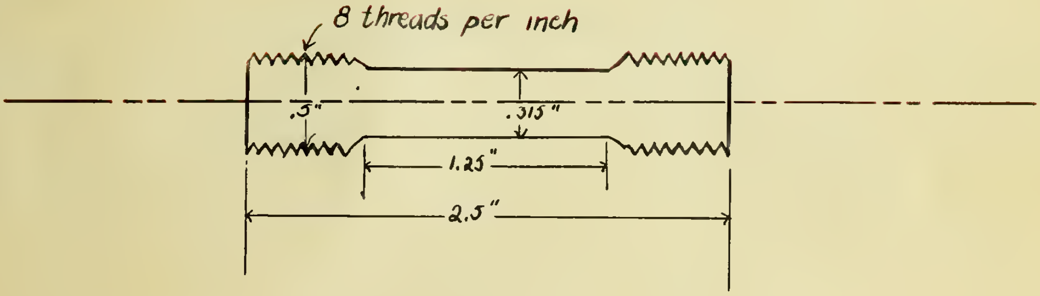
8 threads per inch
.5"
.315"
1.25"
2.5"

Figure 4

IV

THE MICRO-PHOTOGRAPHIC ANALYSIS

Micro sections shown in this thesis are made on the cross section of the micro specimens cut from the same bar as the tensile test specimens and subjected to exactly the same treatment. All of them are prepared in as similar and precise a manner as possible. After hand polishing through six grades of Hubert emery cloth the final polish was acquired on tripoli and rouge wheels. The etching reagent was picric acid in ninety five percent alcohol, and the average time of etching was one minute. Picric acid was selected as the most suitable for defining the grain boundaries. Two standard magnifications were employed, sixty, and three hundred sixty diameters. All photographic work was done with a Leitz metallurgical microscope of the Le Chatelier type, fitted with achromatic lenses.

Certain of the sections show signs of discoloration. This is due to the presence of fumes in the atmosphere on the day on which the sections were photographed and, while unfortunate in obliterating detail, is merely incidental and has no bearing on the properties of the metal.

In mounting on the pages the subjects are grouped as follows. First come three photographs of the material before melting in vacuo; then a series of electrolytic irons are shown which are selected as representing the structural changes that take place in all the processes for which tensile data are presented. A supplementary series for the pure carbon alloys

follows. After these a series of micro sections of the specimens 3-39 and 3-31 are arranged for varying heat treatments. All of the corresponding photographs are of the same section and differ in location only by the depth of the oxide film which is formed during heat treatment.

It is believed that these series of section photographs are the most valuable part of the thesis. The opinion is also entertained that they furnish enough conclusive evidence to warrant some precise deductions.

RESULTS

Results of the preliminary series include three sections: one of the iron as deposited and two of the Swedish charcoal iron. The latter are taken at right angles to the fibrous structure and exhibit the usual slag inclosures of wrought iron. The former is unusual in exhibiting crystalline rather than a granular structure. Its precise mode of preparation is not known, but there is no doubt of the authenticity of the specimen both from its general nature and from the honey comb effect which is the cause of the dark spots in the picture.

In the electrolytic series as forged several facts are observable. The crystalline structure is much finer than any of the other series, and the crystals themselves are greatly distorted. This is caused undoubtedly by the mechanical work while the metal is passing through A_3, and, as uniformity is not to be expected in this process, uniformity is not secured in the

micro-structure. The internal equilibrium is not stable as the further results will show, but the structure accompanies maximum mechanical strength.

In the annealed electrolytic iron series uniformity is secured for specimens annealed and cooled longer than twelve hours. It is thought that there exists a stable number of alpha-beta iron nuclei toward which the specimens cooled for twelve hours are approaching and which the others have actually attained. It is the belief of many eminent metallurgists, notably Ewing and Rosenhain, that in absolutely pure metals the grain will not grow even if the material has previously been strained. After the structure has once attained its equilibrium condition this opinion seems to be sustained.

In the preparation of the quenched sections it was noticed that the surface was metallurgically more resistant to abrasion; polished more readily and exhibited a tendency to polish in relief; and that it etched with greater difficulty, showed a tendency to discolor, and could not be etched in a uniform manner. An inspection of the sections will show a slight decrease in grain size. This is accompanied by a slight increase in mechanical strength. The quenching treatments were invariably violent. It may perhaps be possible to imagine that some of the higher allotropic modifications of iron are retained by this method. This will most readily account for the varying hardness which appears in polishing and for the non-uniformity of the sections.

In the carbon series the specimens 3-CO-1 and 3-CO-2 are practically of the same composition as the electrolytic irons,

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the carbon content of the alloys having been reduced by the furnace in melting. Accordingly, to these two specimens the same remarks made under the electrolytic series also apply.

The alloy 3-CO-3 exhibits the general effect of carbon. The pearlite areas are plainly visible in the forged and annealed sections; the figure for the brine quench is distinctly martensitic, and the liquid air quench is decidedly austenitic. This last is unusual for so low a carbon content, and can be ascribed only to the absence of other impurities.

The specimens in the series 3-39 and 3-31 are the most valuable in the thesis. In the annealings below the point A_3 they illustrate precisely the same phenomena as the previous specimens; but for the annealings above the point A_3 the grain structure becomes greatly subdivided and is replaced by a much finer network. Subsequently with the repeated annealings at a lower temperature this finer structure disappears and is replaced by the stable structure and grain size that is characteristic of the long coolings. The explanation of this can perhaps best be given in the words of its promulgators, Carpenter and Stead. (Journal of the Iron and Steel Institute, September,1913).

"The refining of the structure caused by prolonged heating in the region of gamma iron can be explained in accordance with well established facts of the genesis and growth of crystals in the following way:- On heating the iron strip which consists of an aggregate of alpha crystals, above Ac_3, a region is entered in which gamma is the stable phase, and provided it is held there sufficiently long, not only the alpha crystals, but

their nuclei will be destroyed. When the strip is cooled through Ar_3, and again enters the alpha region it is a question whether a large number of alpha nuclei will be formed simultaneously, in which case the crystals that result from them will be both numerous and small, or a small number of nuclei are formed, in which case, provided diffusion can take place with sufficient rapidity, the nuclei will develop into a small number of large crystals, or, of course, any intermediate proportion may be assumed. A reasonable explanation of the fine alpha crystallization resulting from prolonged heating in the gamma region is, therefore, that very numerous alpha nuclei are formed simultaneously with the passing through Ar_3 and accordingly the crystals are small. On the other hand a short heating above Ac_3 may be insufficient to destroy the alpha nuclei, and when the strip cools through Ar_3 a certain number will be present which will act as centers of crystallization, so that, if diffusion is operative, large crystals will grow from these centers. This would account for the coarse crystallization which results from a short heating above Ac_3, and it is the explanation which appears to the authors to be reasonable, and to fit with the observed facts".

It is also reasonable to expect that from analogy with the lower annealings the gamma iron crystals of the metal in so pure a state would assume a stable condition and would not grow with repeated annealings. Experiment seems to prove that this is finer than the beta-alpha structure. It is possible then to formulate the annealing treatment that will be most desirable in promoting maximum strength.

INDEX TO MICRO-PHOTOGRAPHS

Specimen	Magnification	Treatment	Page
Electrolytic	60	As Depoisited	63
Swedish	60	From Plate	
Swedish	360	" "	
3-43	60	As Forged	64
3-45	360	" "	
3-45	60	" "	
3-45	360	As Forged	65
3-47	60	" "	
3-41	60	Annealed 12 hrs	
3-43	60	Annealed 12 hrs	66
3-45	60	" " "	
3-47	60	" " "	
3-34	60	Annealed 24 hrs	67
3-36	60	' " "	
3-38	60	Annealed 48 hrs	
3-43	60	Annealed 48 hrs	68
3-43	60	" " "	
3-45	60	" " "	
3-47	30	Annealed 48 hrs	69
3-48	60	" " "	
3-54	60	Quenched Brine	
3-36	60	Quenched Brine	70
3-37	60	" "	
3-41	360	Quenched Liquid Air	
3-47	60	Quenched Liquid Air	71
3-CO-1	60	As forged	
3-CO-2	60	" "	
3-CO-2	60	As forged	72
3-CO-3	360	" "	
3-CO-3	360	" "	
3-CO-1	60	Annealed 12 hrs	73
3-CO-2	360	" " "	
3-CO-2	360	" " "	
3-CO-3	60	Annealed 12 hrs	74
3-CO-1	60	Annealed 48 hrs	
3-CO-2	60	" " "	

Department of [illegible]

Specimen	Magnification	Treatments	Page
3-CO-3	60	Annealed 12 hrs	75
3-CO-1	60	Quenched Brine	
3-CO-1	60	" "	
3-CO-2	360	Quenched Brine	76
3-CO-3	360	" "	
3-CO-2	60	Quenched Liquid Air	
3-CO-3	360	Quenched Liquid Air	77
3-39	60	As forged	
	60	Furnace cooled 680	
3-39	60	Furnace cooled 800	78
	60	" " 950	
	60	" " 1000	
3-39	360	Furnace cooled 1000	79
	60	" " 1060	
	60	" " 912	
3-39	360	Furnace cooled 800	80
	60	" " 700	
	60	Quenched Brine	
3-39	60	Annealed 6 hrs	81
	60	Annealed 48 hrs	
3-31	60	As forged	
3-31	60	Furnace cooled 700	82
	60	" " 800	
	60	" " 800	
3-31	60	Furnace cooled 900	83
	60	" " 950	
	60	Annealed 48 hrs	

[illegible]

Specimen	Treatment	Page
3-34	Annealed 24 hrs	67
	Quenched Brine	69
3-36	Annealed 24 hrs	67
3-37	Quenched Brine	70
3-38	Annealed 48 hrs	67
3-41	Annealed 12 hrs	65
	Quenched Liquid Air	70
3-43	As forged	64
	Annealed 12 hrs	66
	Annealed 48 hrs	68
3-45	As forged	64-65
	Annealed 12 hrs	66
	Annealed 48 hrs	68
3-47	As forged	65
	Annealed 12 hrs	66
	Annealed 48 hrs	69
	Quenched Liquid Air	71
3-48	Annealed 48 hrs	69

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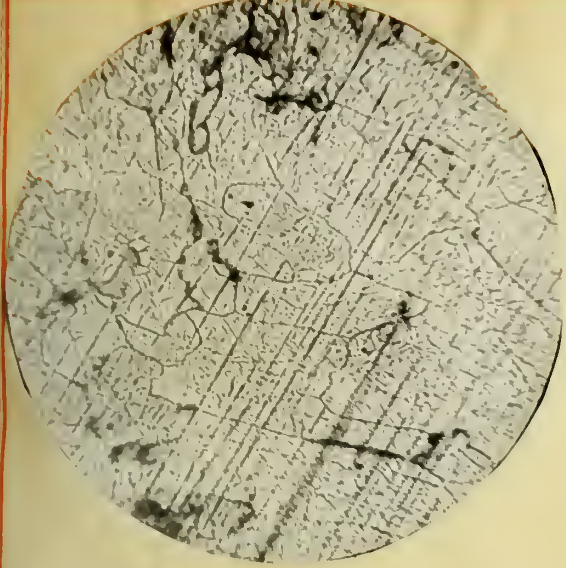
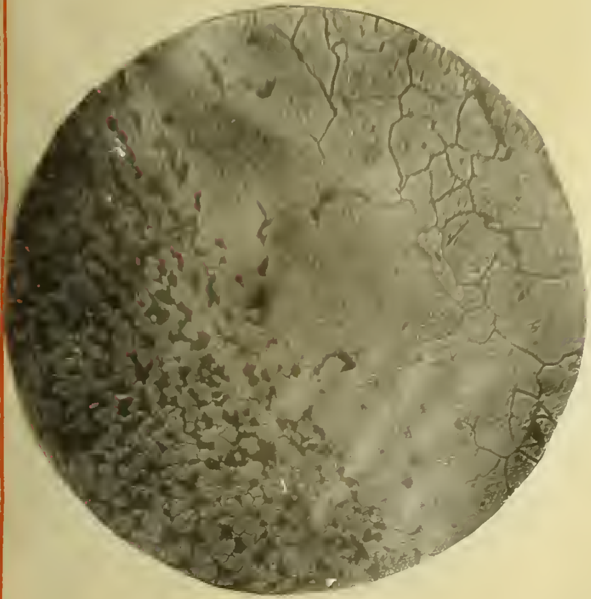
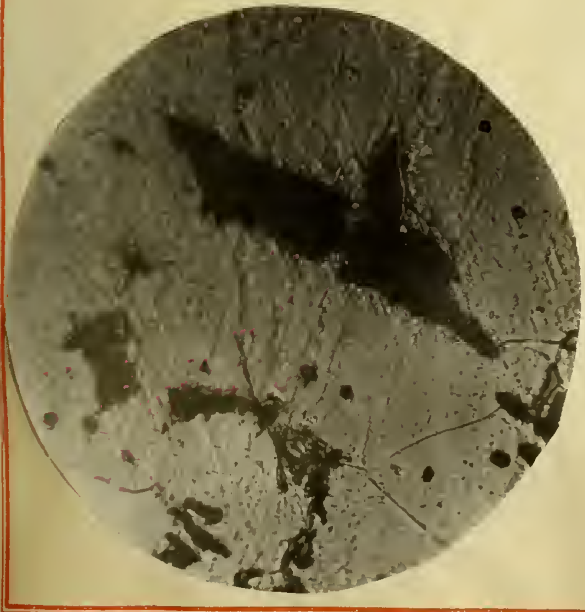


Electro-lytic Iron as
deposited
60 diameters

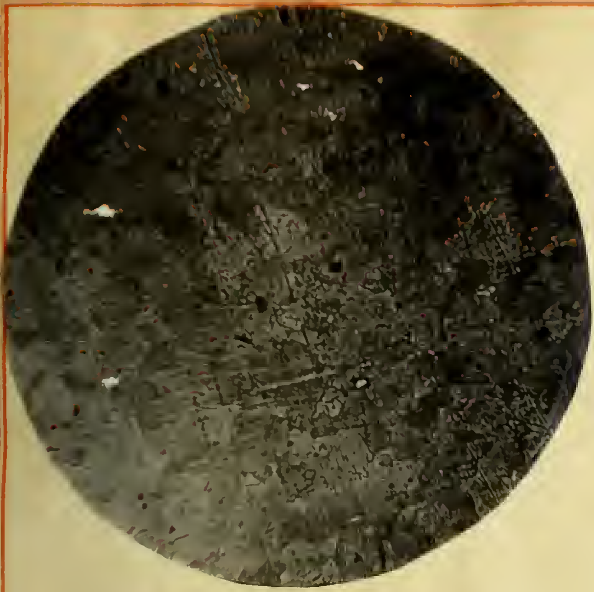


Swedish Iron as cut from
cathode plate
60 diameters

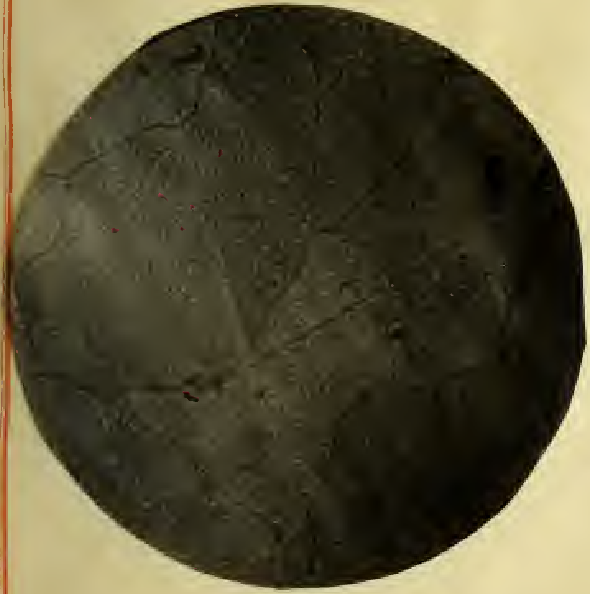


500 diameters

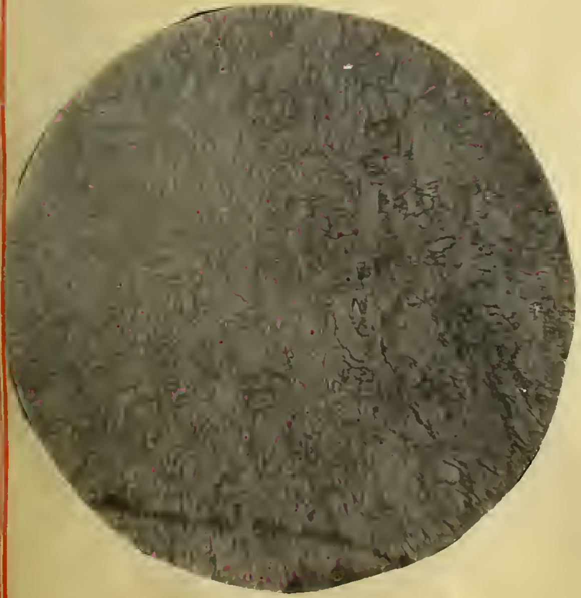
VACUUM FURNACE IRON



As Forged 3-13
60 diameters

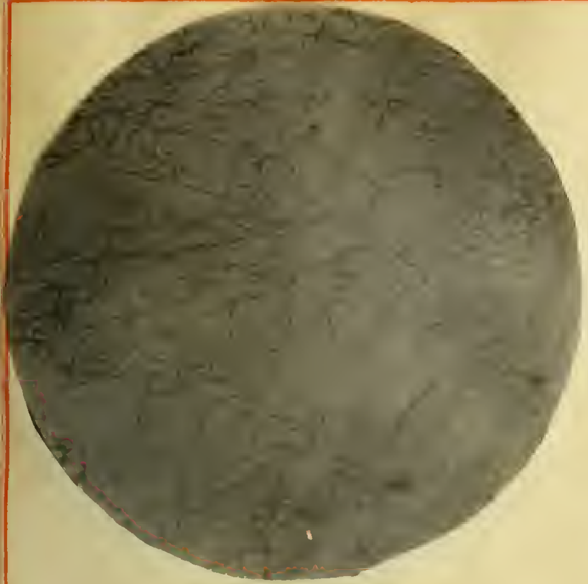


360 diameters

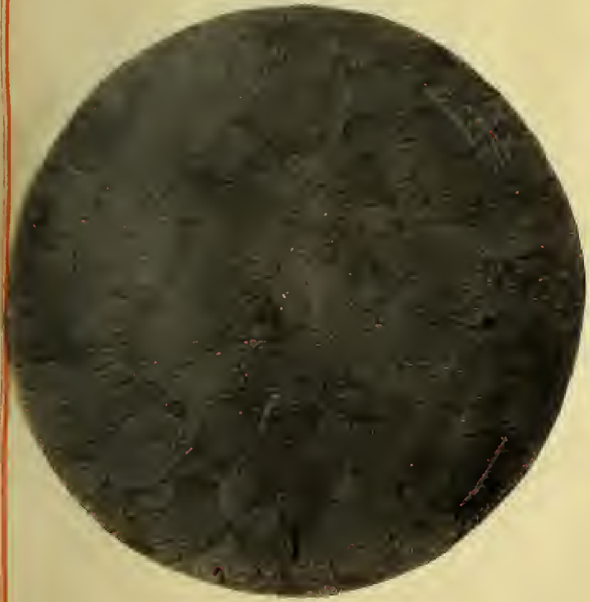


As Forged 3-45
60 diameters

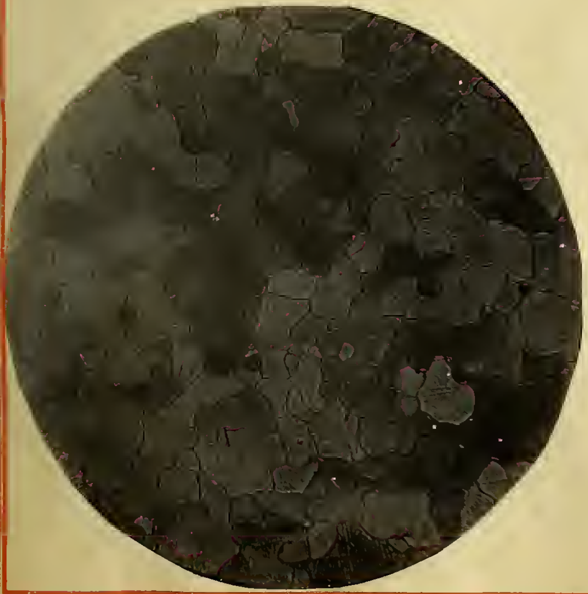
[illegible]



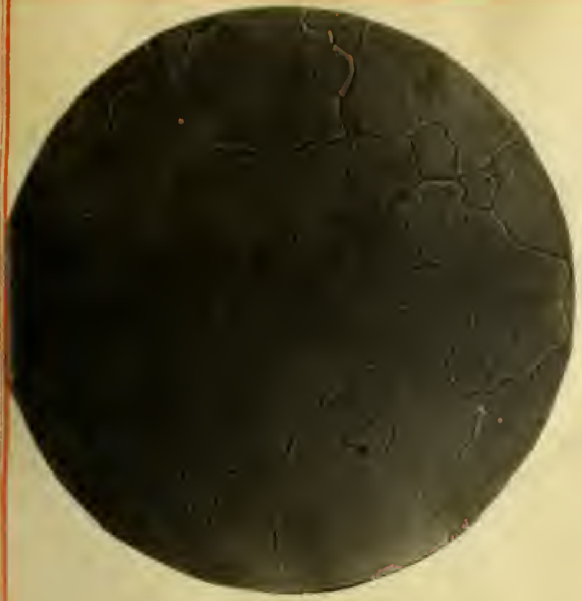
As Forged 3-45
360 diameters



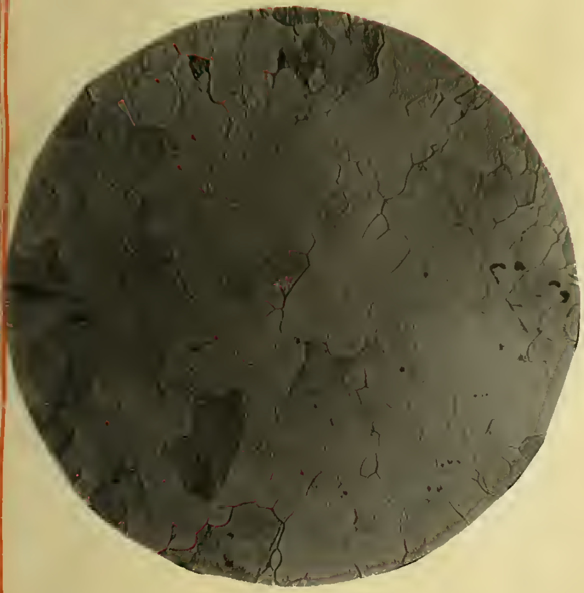
As Forged 3-47
50 diameters



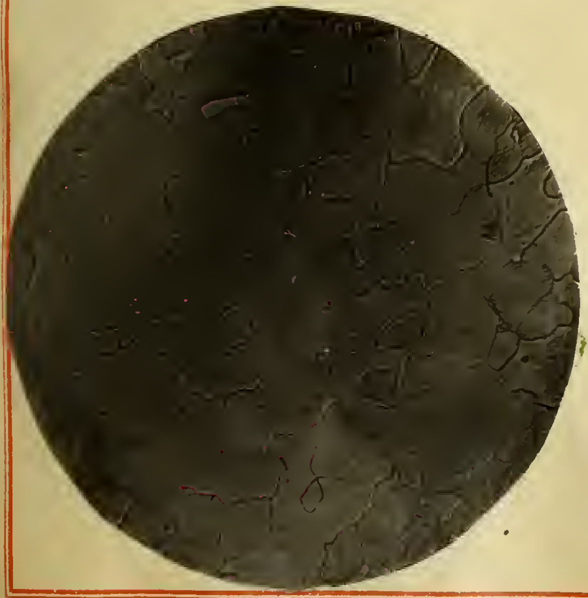
Annealed 900.Cooled 12 hrs
3-41
50 diameters



Annealed 900.Cooled 12 hrs
3-43
60 diameters

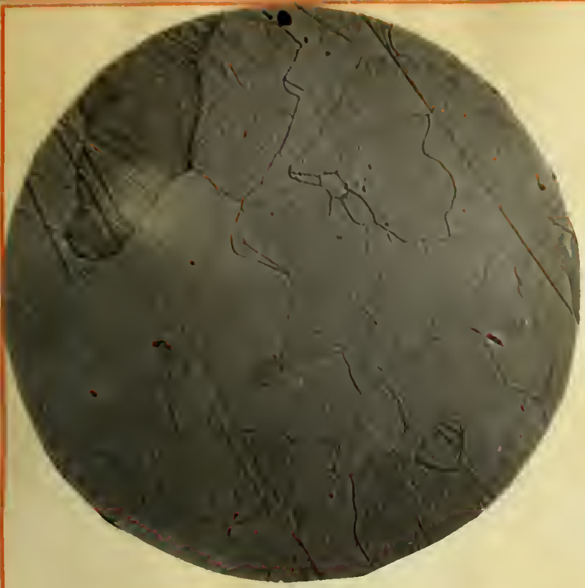


Annealed 900.Cooled 12 hrs
3-45
60 diameters

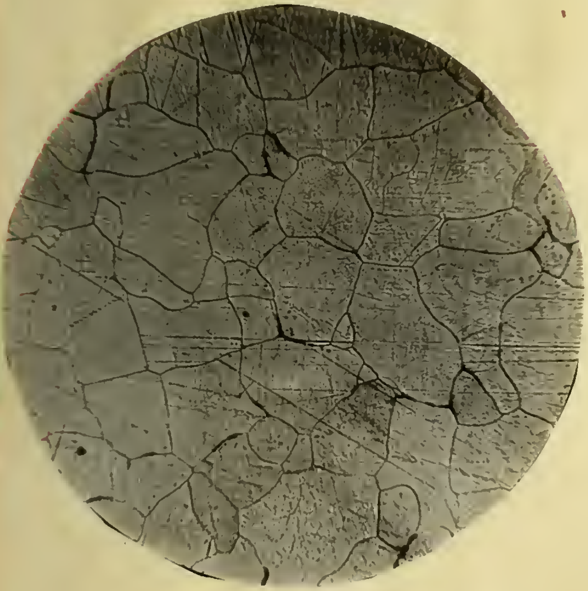


Annealed 900.Cooled 12 hrs
3-47
60 diameters

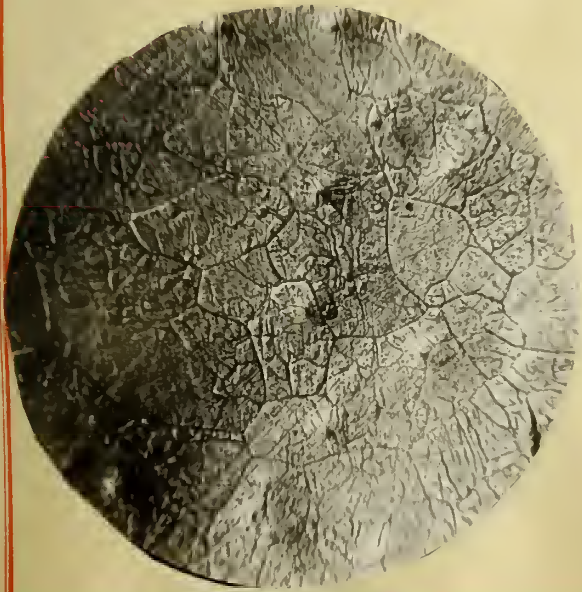
[illegible]



Annealed 900.Cooled 24 hrs
3-34
60 diameters

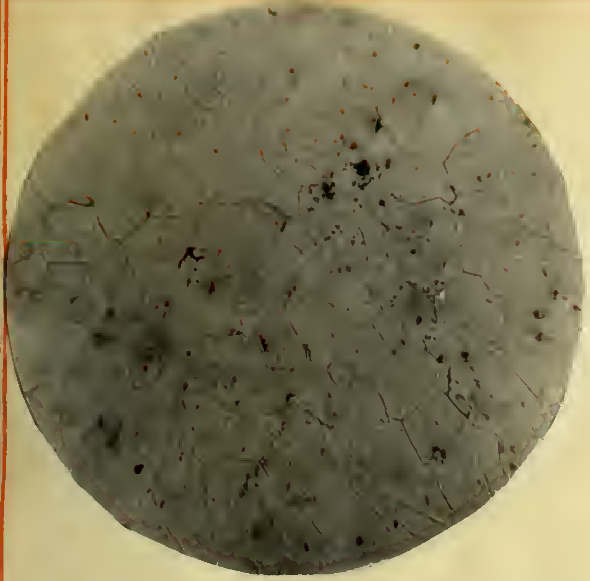


Annealed 900.Cooled 24 hrs
3-36
60 diameters

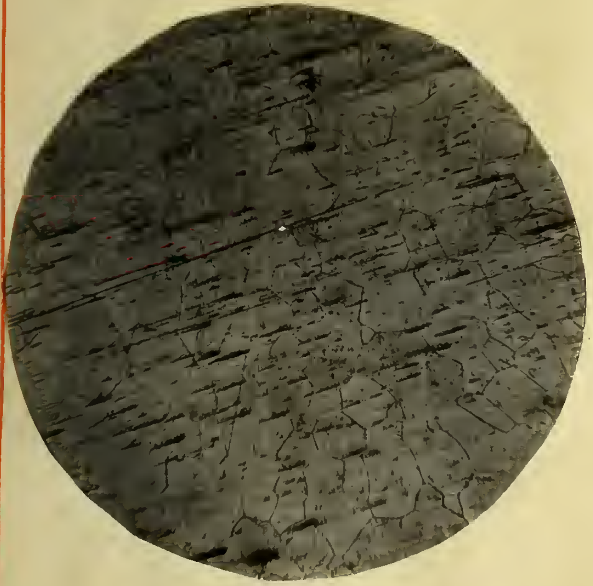


Annealed 900.Cooled 48 hrs
3-38
60 diameters

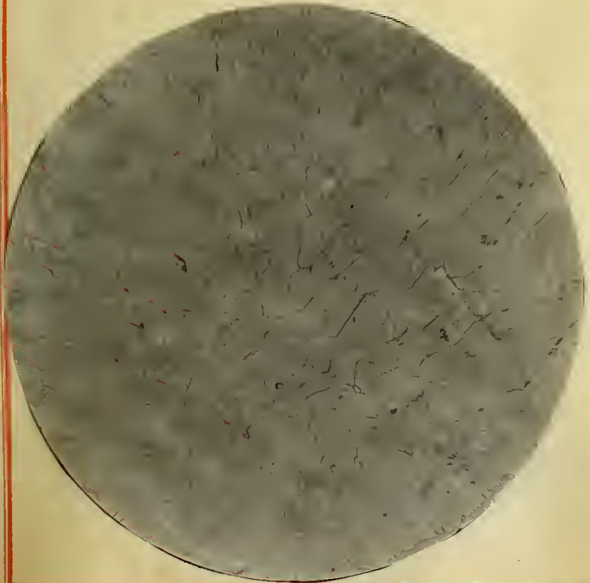
[illegible]



Annealed 900.Cooled 48 hrs
3-45
50 diameters

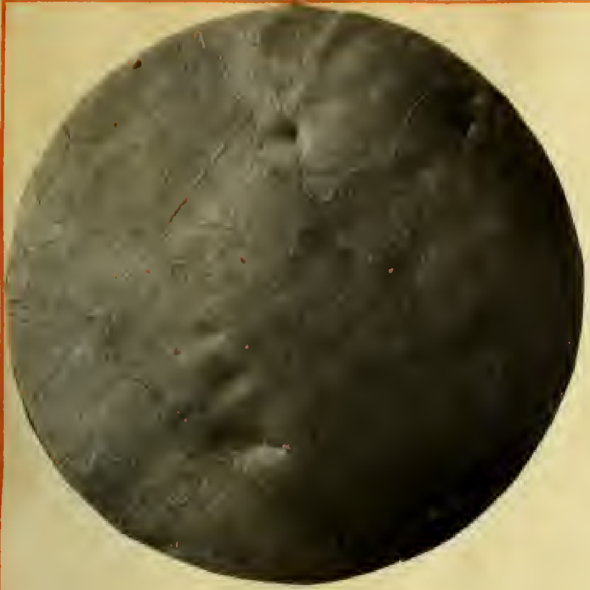


Annealed 900.Cooled 48 hrs
3-43
50 diameters

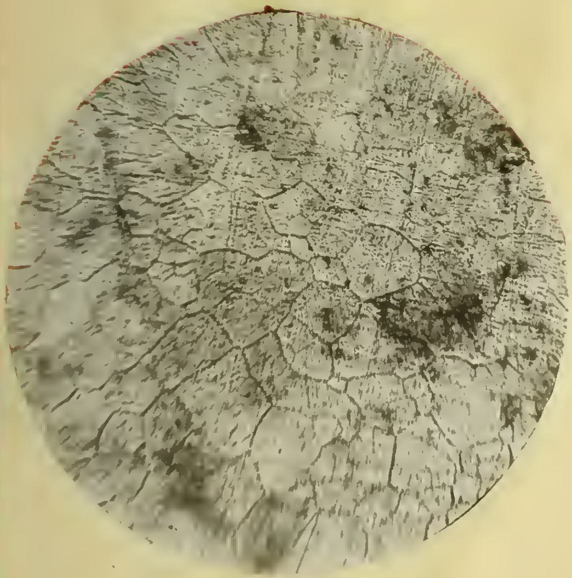


Annealed 900.Cooled 48 hrs
3-45
50 diameters

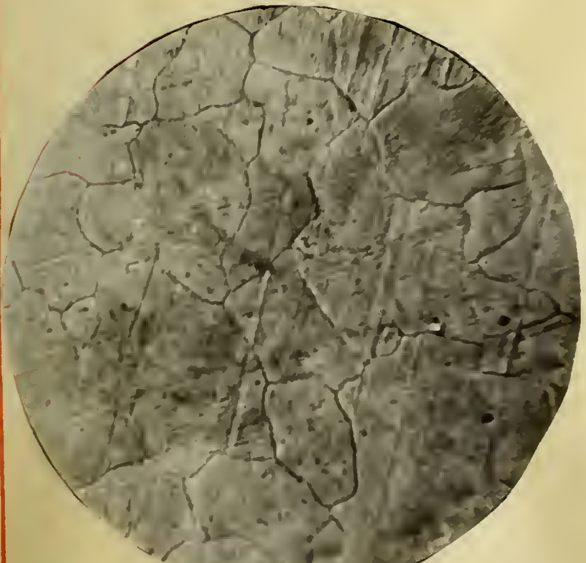
[illegible]



Annealed 900.Cooled 48 hrs
3-47
60 diameters

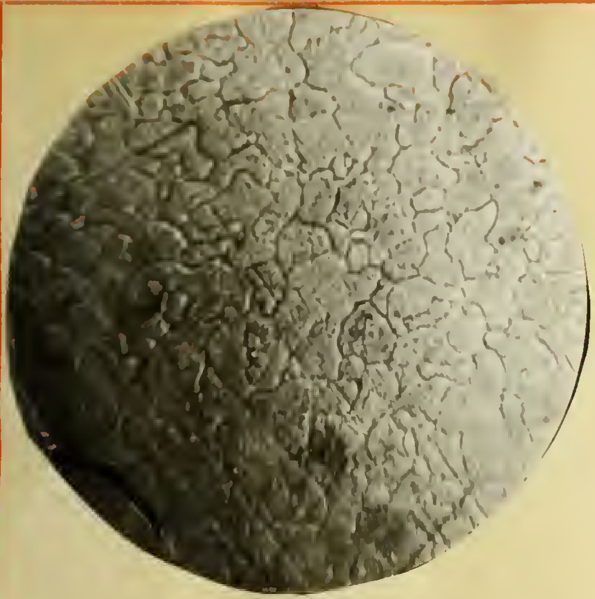


Annealed 900.Cooled 48 hrs
3-48
60 diameters

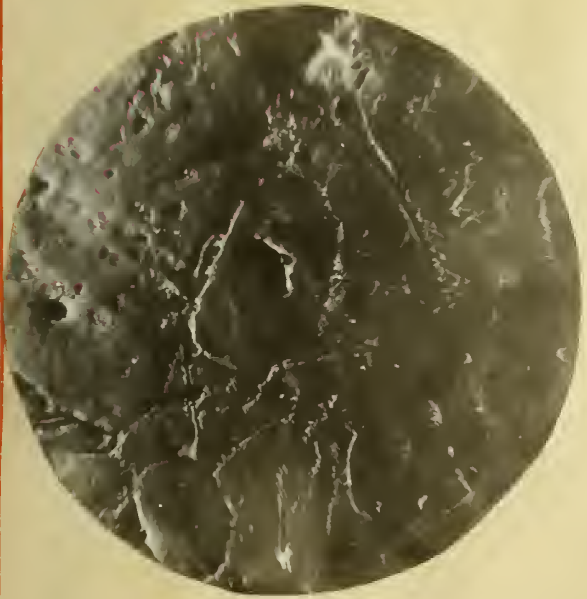


Quenched 1000.Brine (-21)
3-34
60 diameters

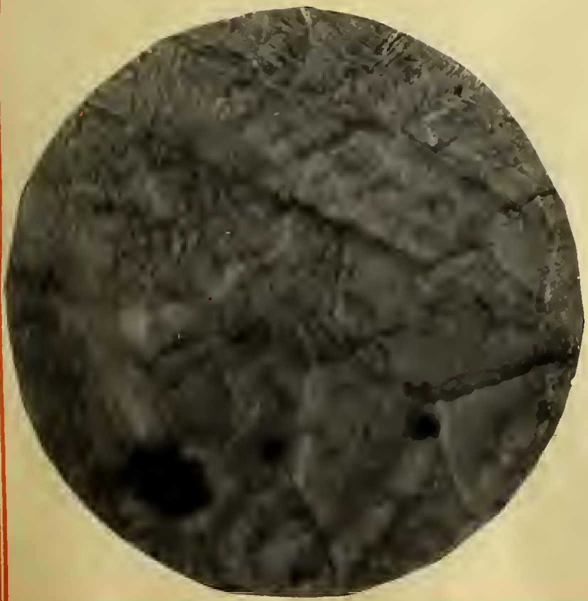
[illegible]



Quenched 1000.Brine (-21)
3-36
60 diameters



Quenched 1000.Brine (-21)
3-37
60 diameters



Quenched 1000.Liquid Air
3-41
360 diameters

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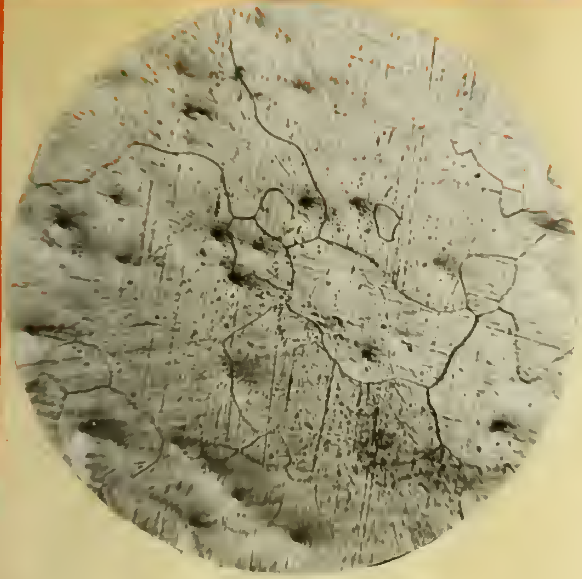
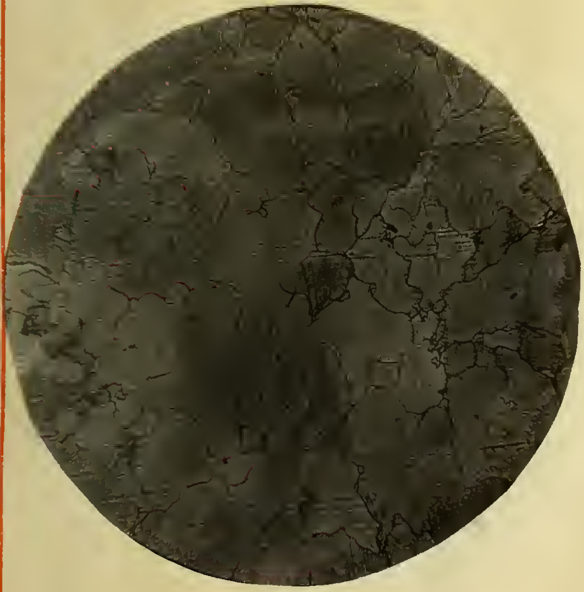
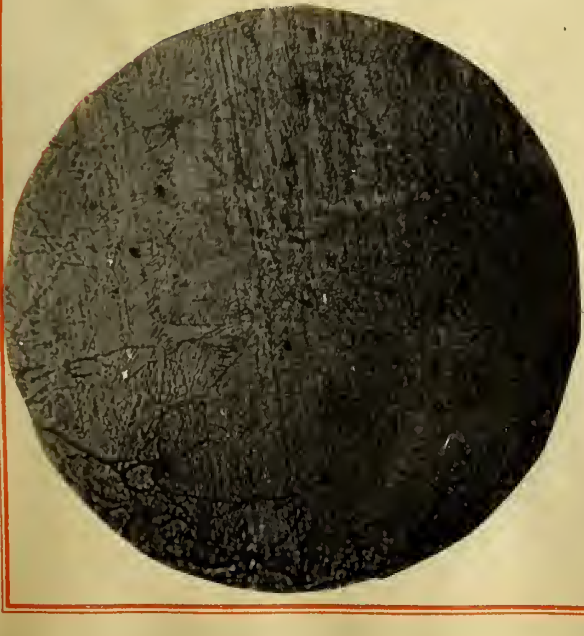


Quenched 1000.Liquid Air
3-47
60 diameters

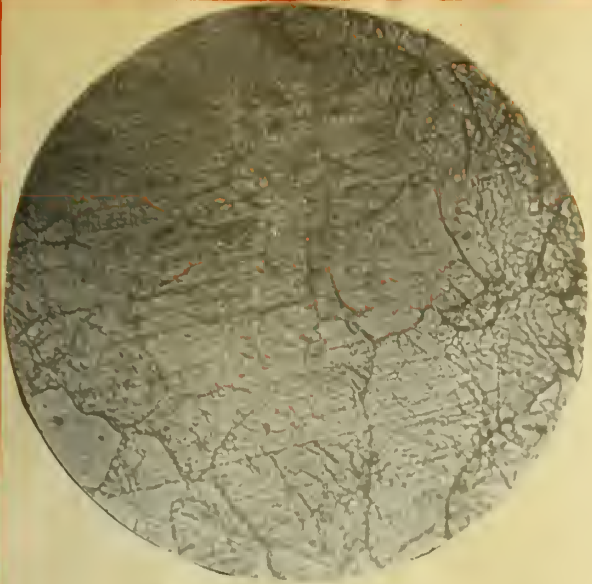
CARBON ALLOYS



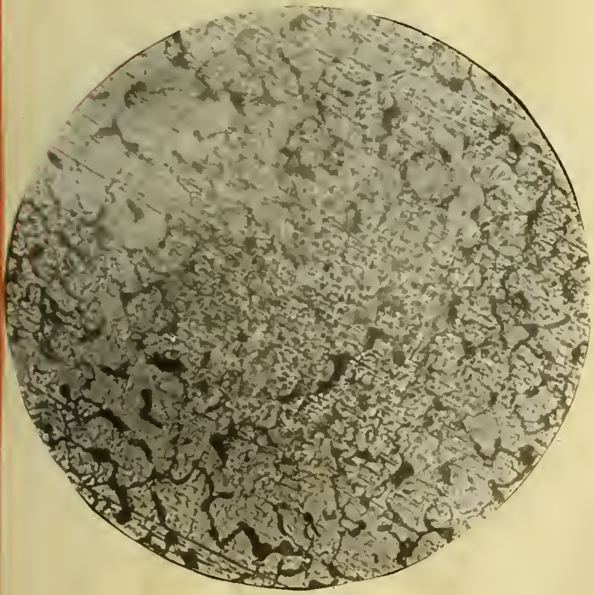
As forged. 3-CO-1
~~3~~60 diameters



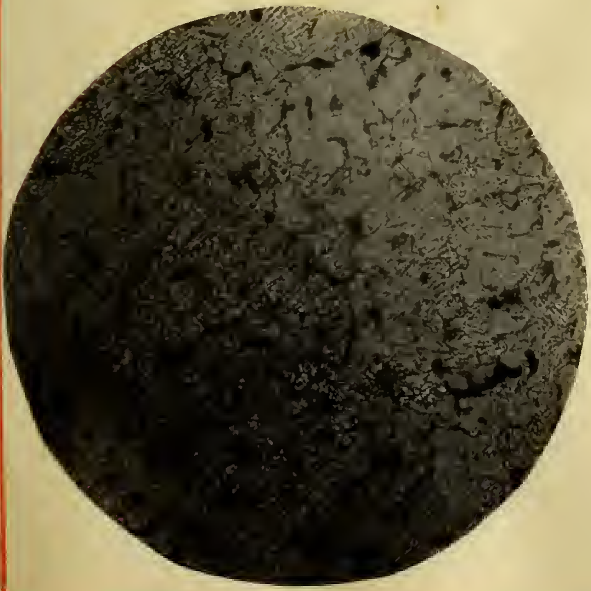
As forged.3-CO-2
60 diameters



As forged 3-C0-2
60 diameters

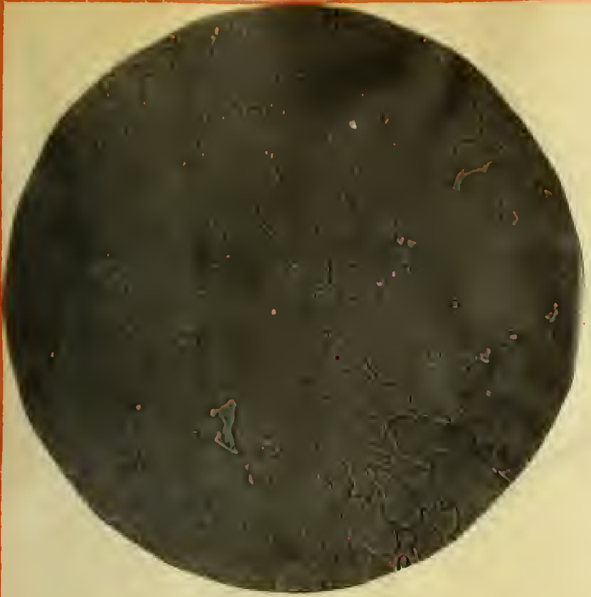


As forged 3-C0-3
360 diameters

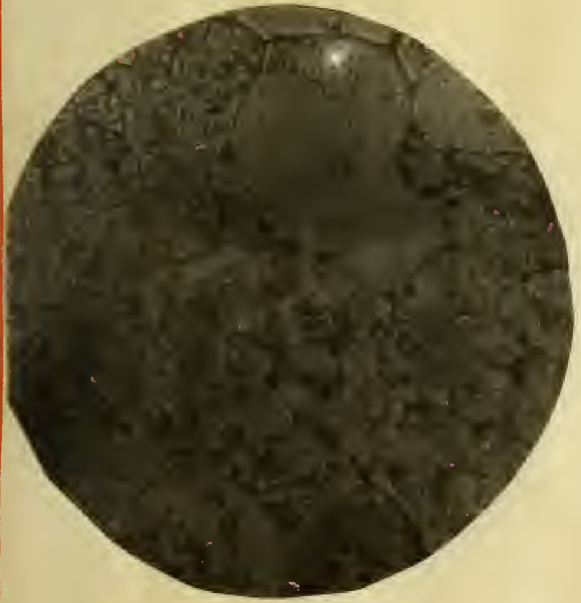


As forged 3-C0-3
360 diameters

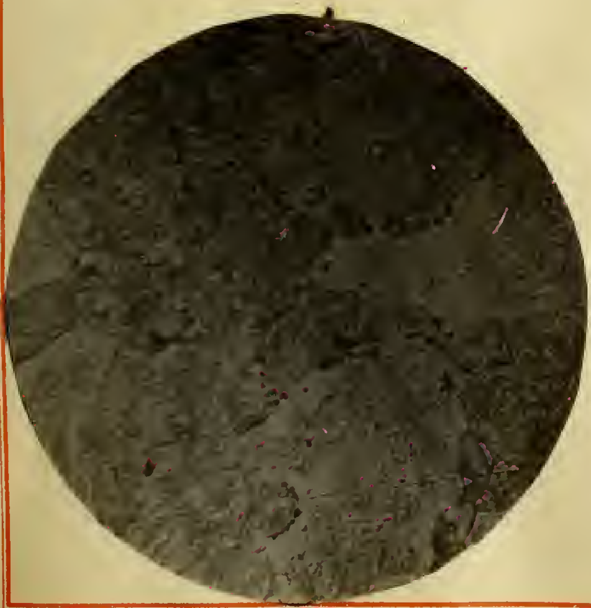
[illegible]



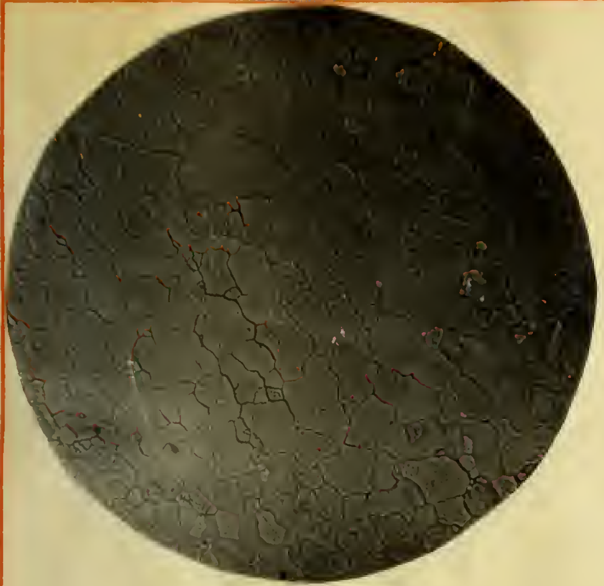
Annealed 900.Cooled 12 hrs
3-C0-1
60 diameters



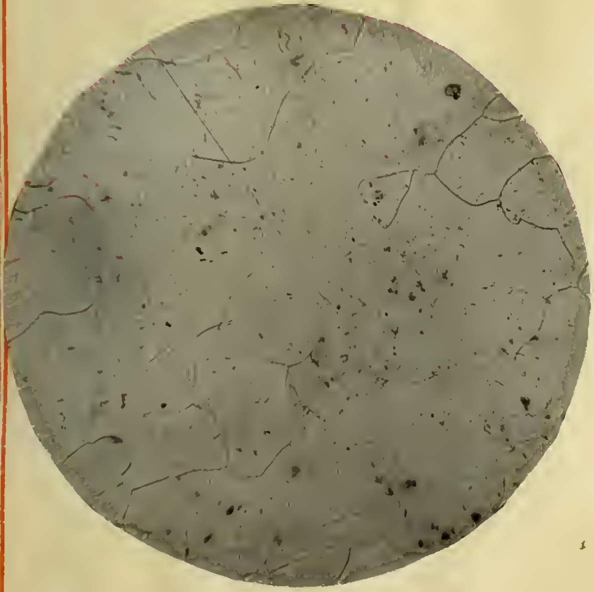
Annealed 900.Cooled 12 hrs
3-C0-2
360 diameters



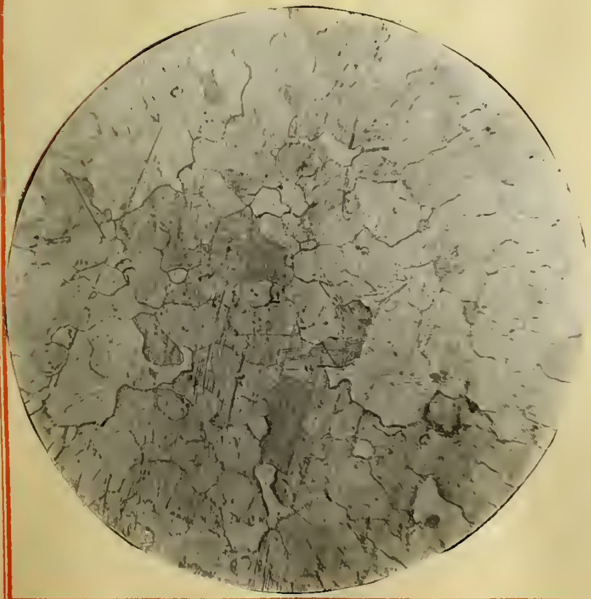
360 diameters



Annealed 900.Cooled 15 hrs
3-CO-3
60 diameters

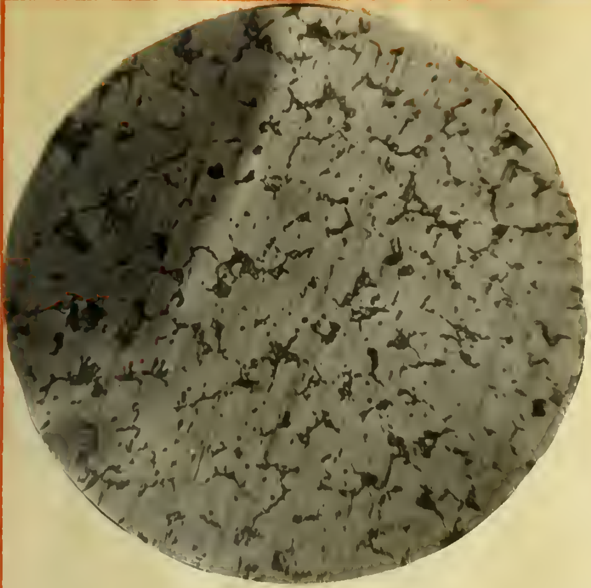


Annealed 900.Cooled 48 hrs
3-CO-1
60 diameters

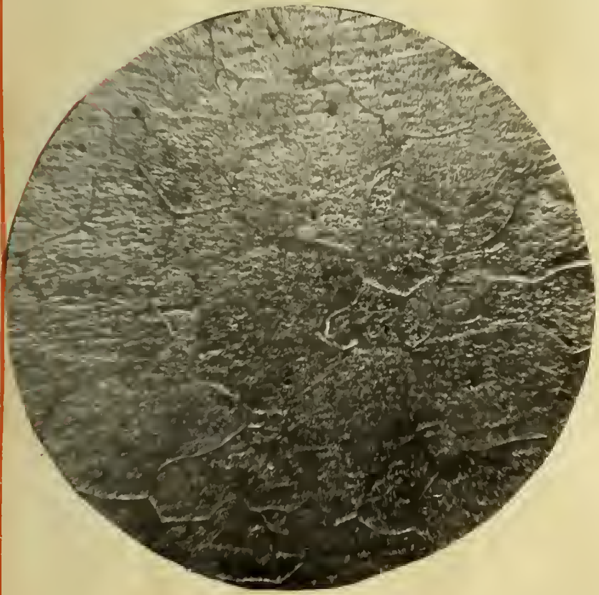


Annealed 900.Cooled 48 hrs
3-CO-2
60 diameters

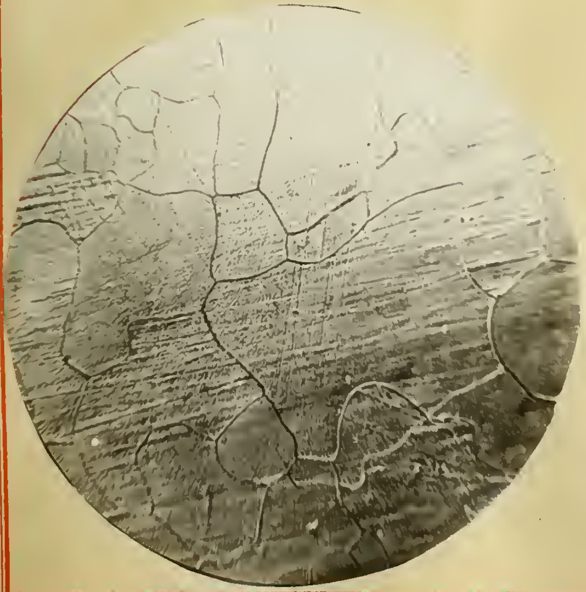
[illegible]



Annealed 900.Cooled 48 hrs
3-CO-3
60 diameters

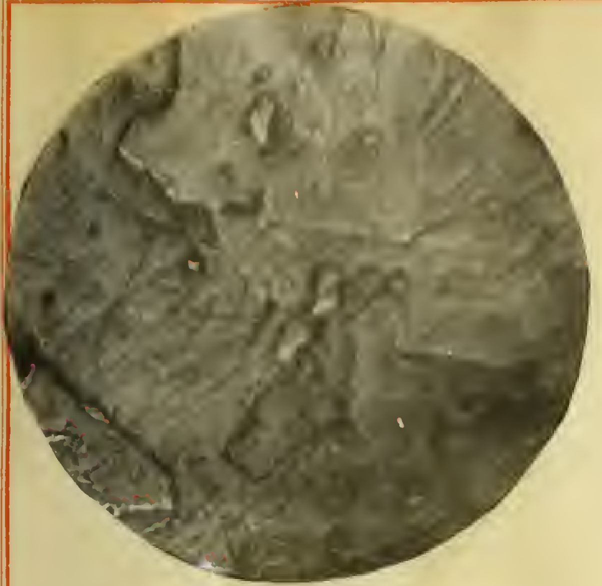


Quenched 1000.Brine (-21)
3-CO-1
60 diameters

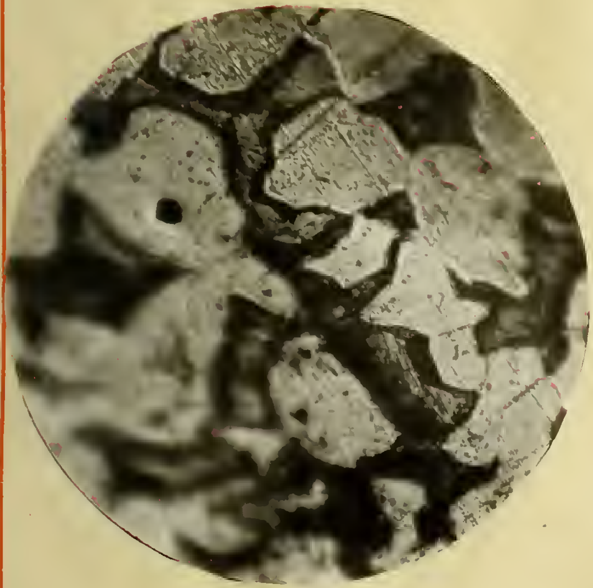


60 diameters

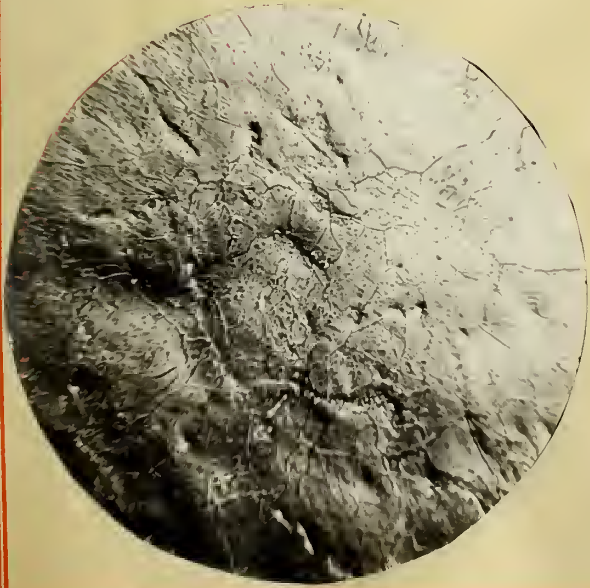
[illegible]



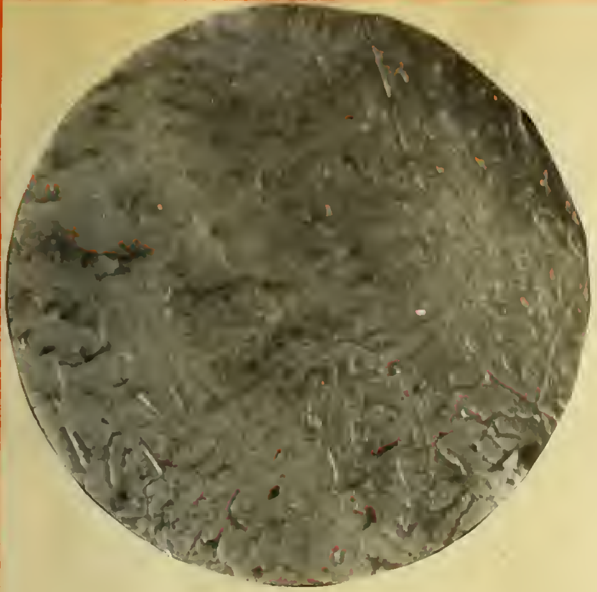
Quenched 1000.Brine (-21)
3-C0-2
360 diameters



Quenched 1000.Brine (-21)
3-C0-3
360 diameters

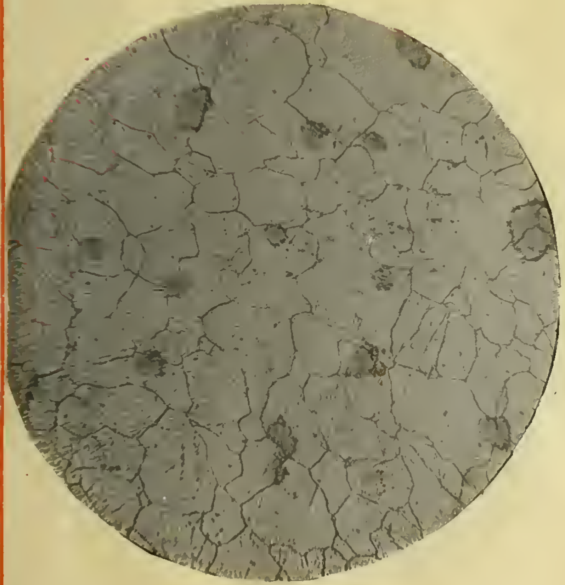


Quenched 1000.Liquid Air
3-C0-2
60 diameters

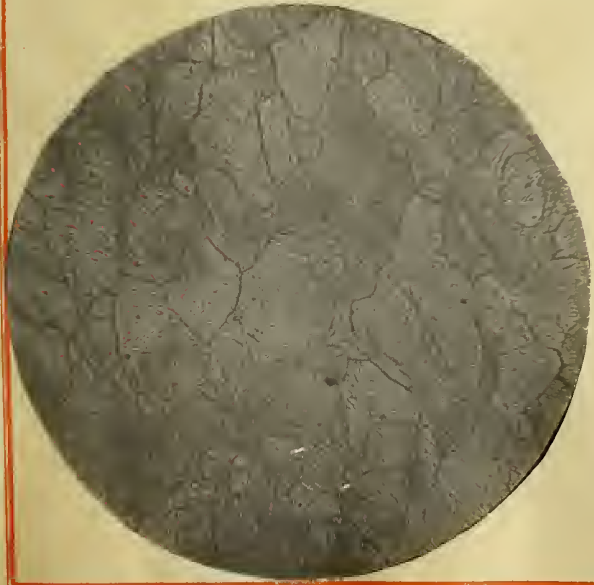


Quenched 1000.Liquid Air
3-CO-3
350 diameters

SERIES 3-39

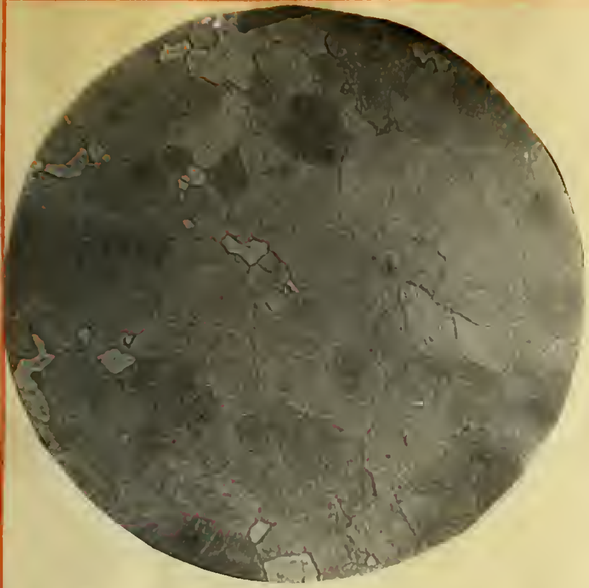


As forged
60 diameters

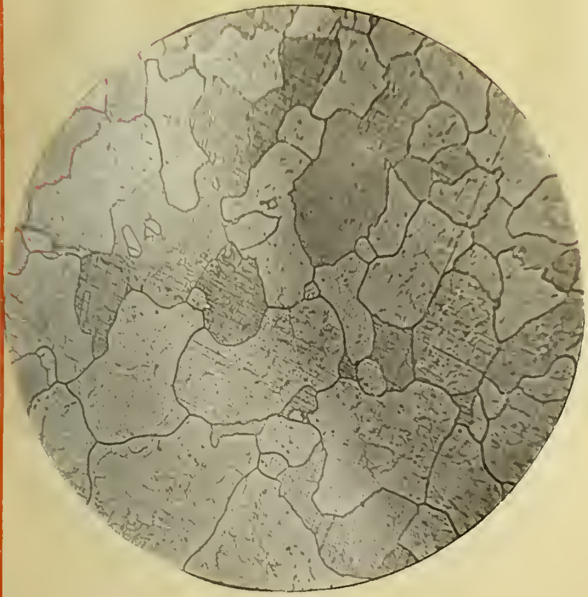


Annealed 680.Furnace Cooled
60 diameters

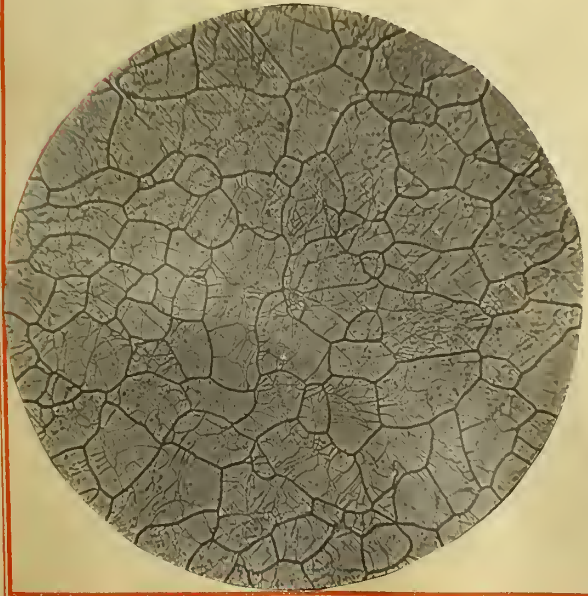
[illegible]



Annealed 800.Furnace Cooled
60 diameters



Annealed 950.Furnace Cooled
60 diameters



Annealed 1000.Furnace Cooled
60 diameters

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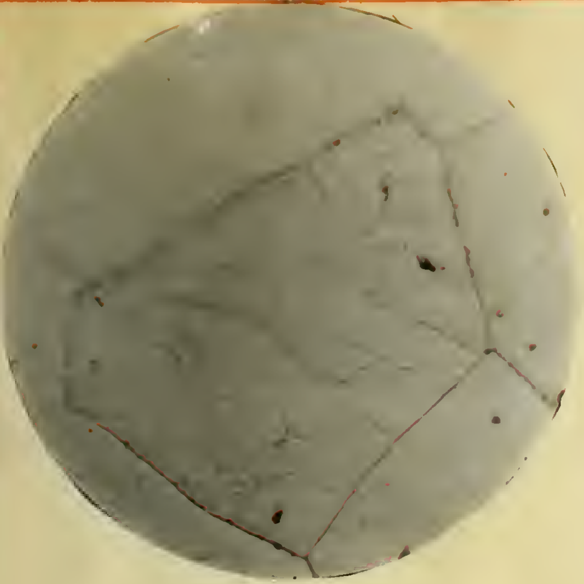
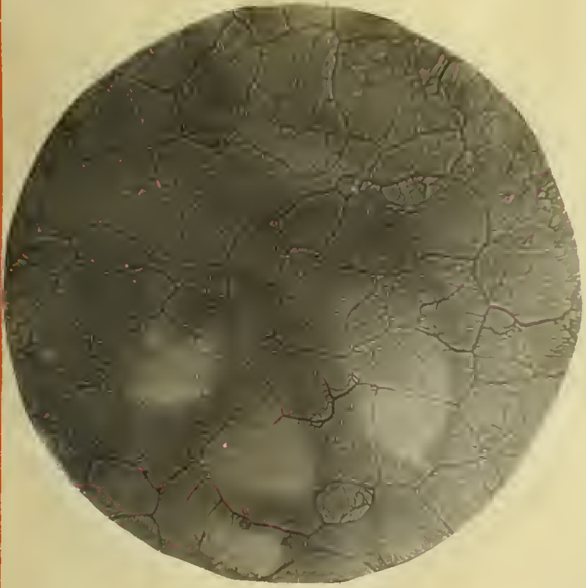
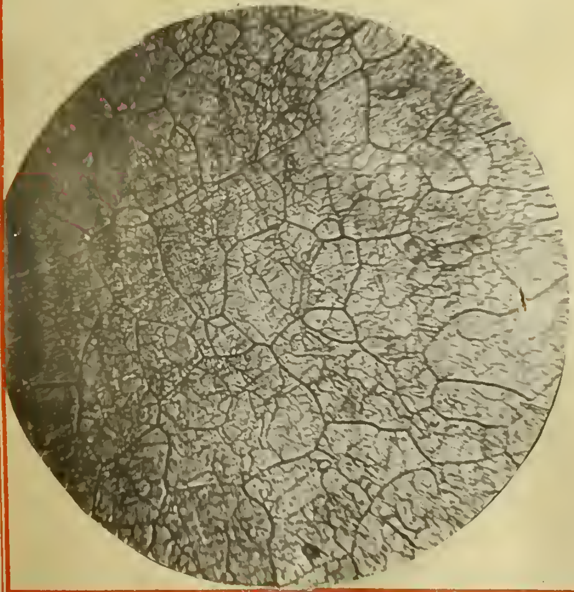


360 diameters



Annealed 1060.Furnace Cooled
50 diameters



Annealed 912.Furnace Cooled
50 diameters

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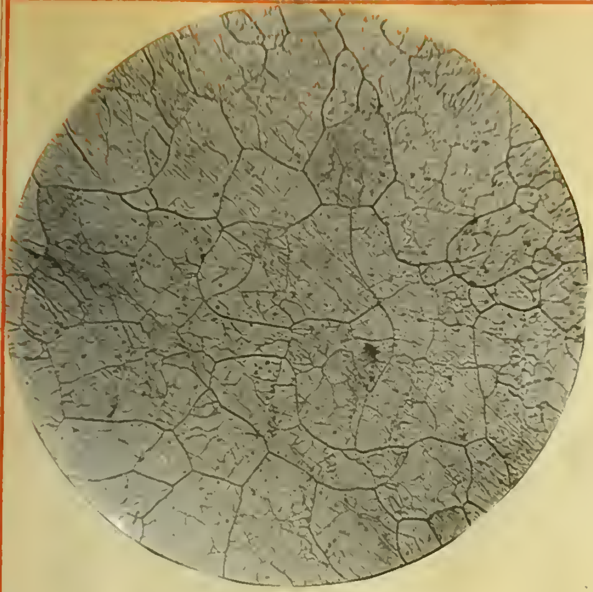
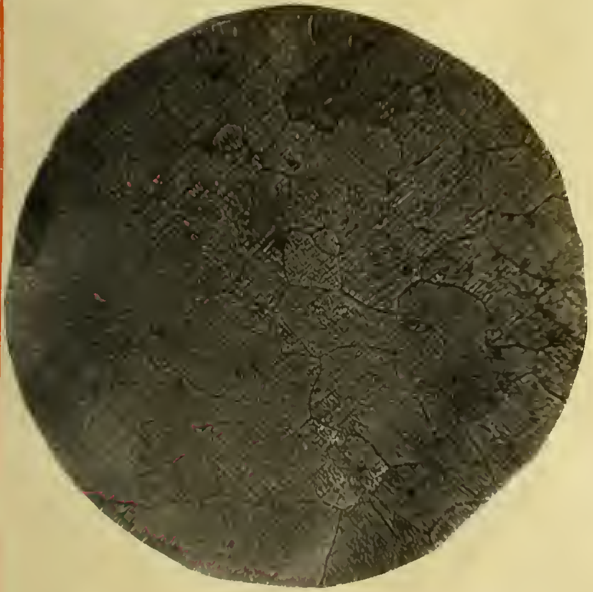
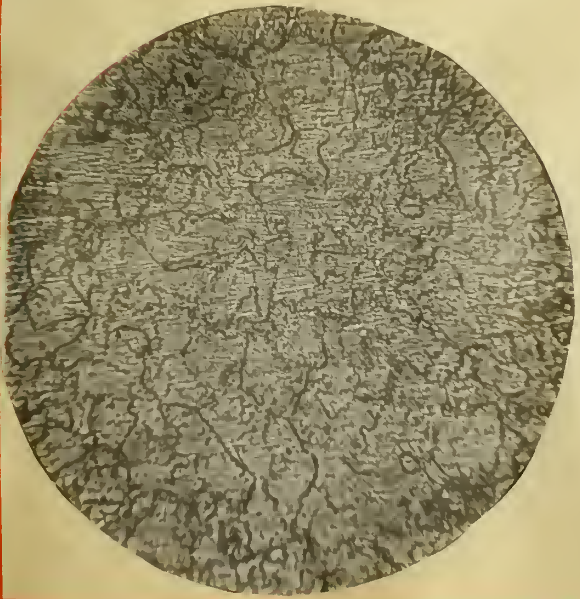


Annealed 800.Furnace Cooled
360 diameters



Annealed 700.Furnace Cooled
60 diameters



Quenched 1000.Brine (-21)
60 diameters

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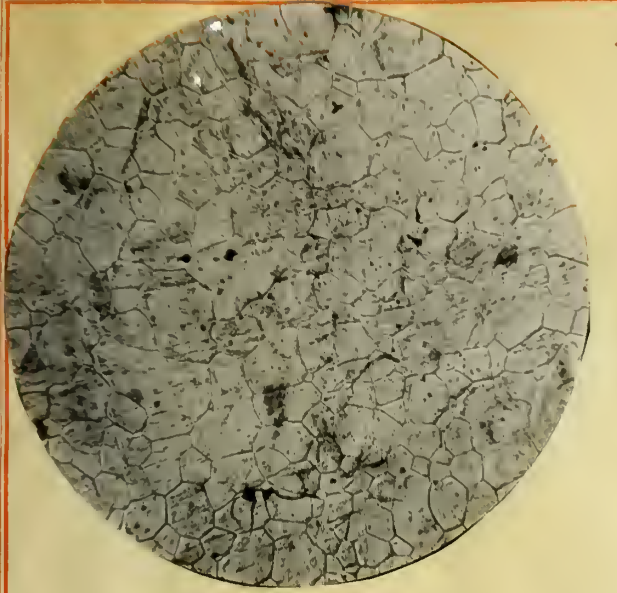
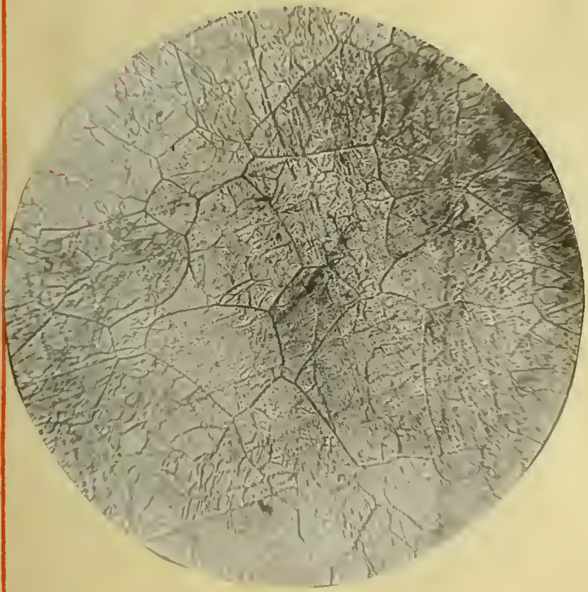
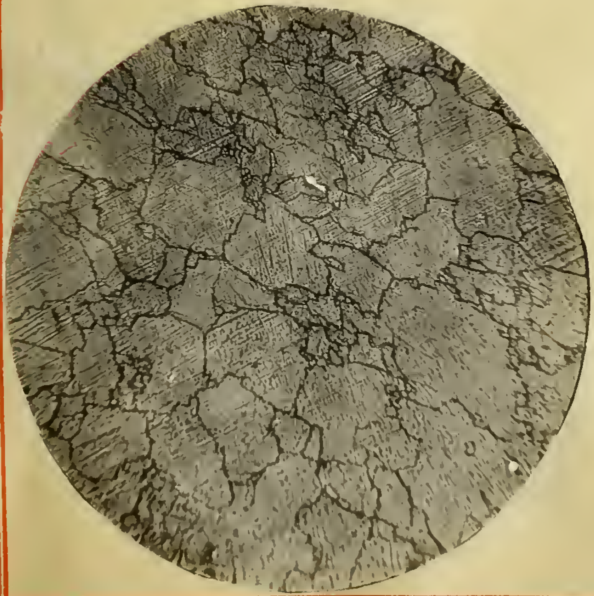


Annealed 900.Cooled 6 hrs
60 diameters



Annealed 900.Cooled 48 hrs
60 diameters

SERIES 3-31



As forced
60 diameters

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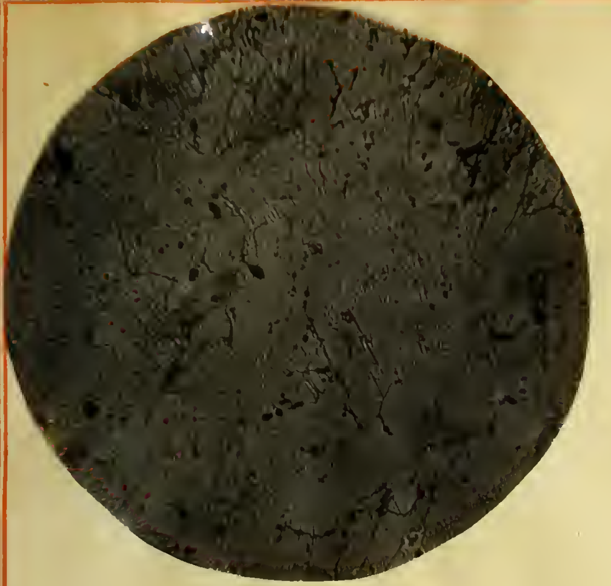
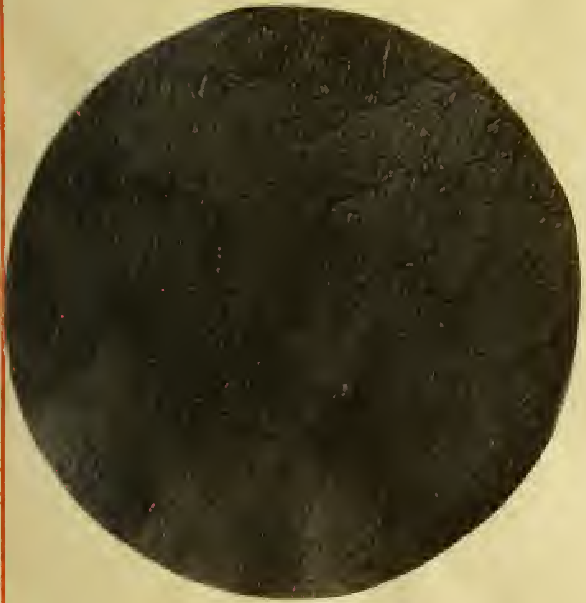
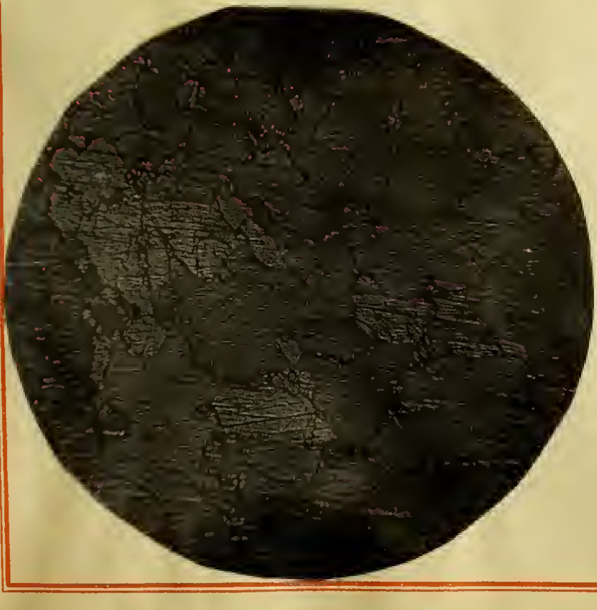


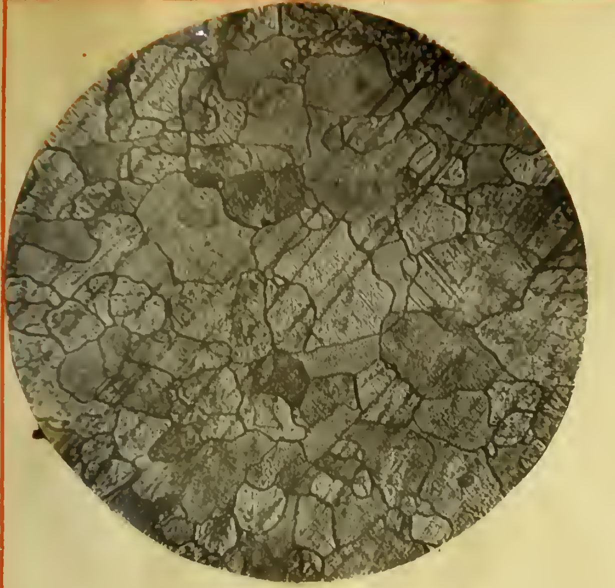
Annealed 700.Furnace Cooled
60 diameters



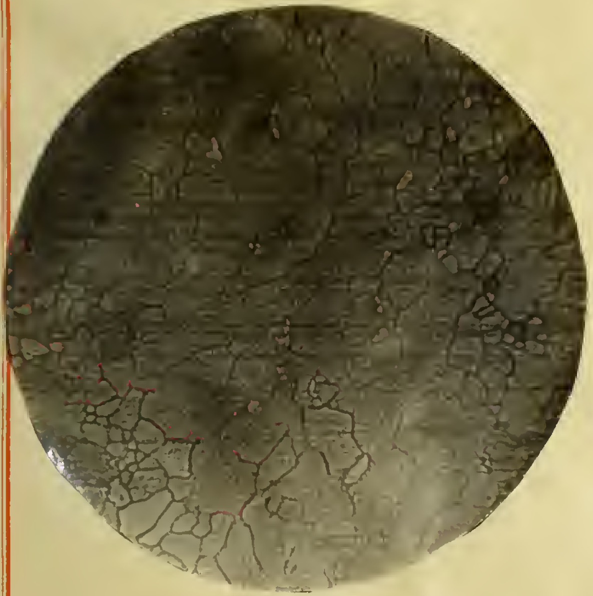
Annealed 800.Furnace Cooled
60 diameters



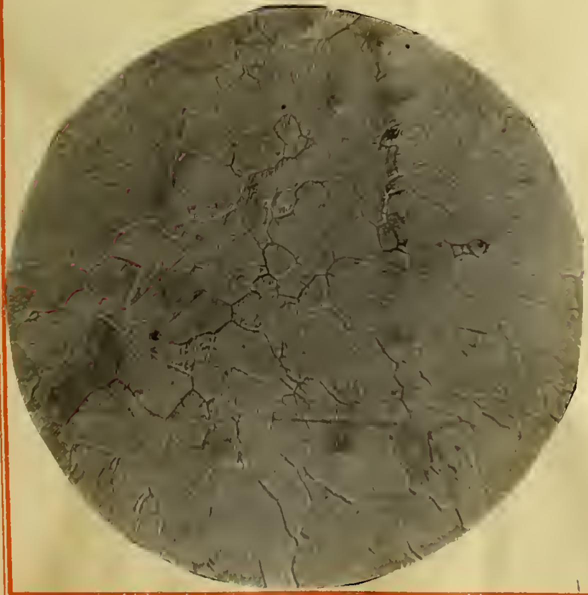
60 diameters



Annealed 900.Furnace Cooled
60 diameters



Annealed 950.Furnace Cooled
60 diameters



Annealed 900.Cooled 48 hrs
60 diameters

V

SUMMARY

It is hoped that to the numerical data presented the following qualitative facts may be added.

Pure iron in cooling from its melting point to ordinary temperatyres passes through two transition points which this thesis will approximately locate.

Iron of the purity of the samples used does not show crystalline growth beyond a certain maximum stable equilibrium condition regardless of the number or methods of annealing, provided it does not pass beyond the beta stage. This fact may be stated variously that pure iron which has not previously been strained or from which the state of strain has been removed does not undergo any increase in grain size from any manner of annealing below the temperature of A_3.

The heat treatment for the finest crystalline structure and the accompanying maximum mechanical strength should be somewhat as follows:- the iron should be annealed for a long period at a temperature slightly above the A_3 transition point the region where the gamma modification is stable; it should next be cooled rapidly either by quenching or air blast to atmospheric, and then again annealed for a very long period at a temperature not exceeding 600 C. This is very similar to the empirical treatment given silicon steel.

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